U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

PB-270 897

# Destroying Chemical Wastes in Commercial Scale Incinerators, Facility Report No. 6. Rollins Environmental Service, Inc., Deer Park, Texas

TRW Defense and Space Systems Group, Redondo Beach, Calif.

Prepared for

Environmental Protection Agency, Washington, D.C.

1977

**BIBLIOGRAPHIC DATA SHEET**

1. Report No.

2.

3. Recipient's Accession No.

4. Title and Subtitle

DESTROYING CHEMICAL WASTES IN COMMERCIAL SCALE INCINERATORS
Facility Report No. 6 - Rollins Environmental Service, Inc.

5. Report Date

6.

7. Author(s) D. Ackerman, J. Clausen, R. Johnson, R. Tobias, C. Zee J. Adams, J. Harris, P. Levins, J. Stauffer, K. Thrun, L. Woodland

8. Performing Organization Rept. No.

9. Performing Organization Name and Address

TRW Defense and Space Systems Group
One Space Park, Redondo Beach, California 90278

10. Project/Task/Work Unit No.

11. Contract/Grant No.

68-01-2966

12. Sponsoring Organization Name and Address

U.S. Environmental Protection Agency
Office of Solid Waste Management Programs
Washington, D. C. 20460

13. Type of Report & Period Covered

Facility test report

14. [illegible]

15. Supplementary Notes

16. Abstracts

Incineration tests were conducted at Rollins Environmental Services, Inc., Deer Park, Texas, to determine the effectiveness of thermally destroying two selected industrial wastes: PCB-containing capacitors and nitrochlorobenzene waste (NCB). Analysis of combustion gas samples indicated destruction efficiencies of over 99.999 percent for each waste constituent. Some PCBs were detected in the ash when whole capacitors were incinerated in the rotary kiln, but not when hammermilled capacitors were burned. Standard EPA Method 5 tests were performed on stack emission to determine particulate loading and composition. Estimated cost to hammermill and incinerate 5000 metric tons of waste capacitors per year is $3.65 capital investment and an operating cost of $751/metric ton. Cost of incinerating 4540 metric tons/year of NCB was estimated to be $2.82 million capital and $283/metric ton operating costs.

17. Key Words and Document Analysis. 17a. Descriptors

Waste Disposal
Industrial Wastes
Incinerators
Field Tests
Gas Sampling
Chemical Analysis
Cost Analysis

17b. Identifiers/Open-Ended Terms

Rollins Environmental Services, Inc.
Rotary Kiln Incinerator
Liquid Injection Incineration
Polychlorinated Biphenyls
Nitrochlorobenzine

17c. COSATI Field/Group 07-01, 14-02, 14-01

18. Availability Statement

19. Security Class (This Report) UNCLASSIFIED

20. Security Class (This Page) UNCLASSIFIED

21. No. of Pages 173

22. Price [illegible]

This report has been reviewed by the U.S. Environmental Protection Agency and approved for publication. Its publication does not signify that the contents necessarily reflect the views and policies of the U.S. Environmental Protection Agency, nor does mention of commercial products constitute endorsement or recommendation for use by the U.S. Government.

An environmental protection publication (SW-122c.5) in the solid waste management series.

## FOREWORD

The tests described in this report are part of a program designed to evaluate the environmental, technical, and economic feasibility of disposing of industrial wastes via incineration. This objective is being pursued through a series of test burns conducted at commercial incinerators and with real-world industrial wastes. Seven incineration facilities and fourteen different industrial wastes will be tested under this program. The incineration facilities were selected to represent the various design categories which appear most promising for industrial waste disposal. The wastes were selected on the basis of their suitability for disposal by incineration and their environmental priority.

This report describes the test conducted at Rollins Environmental Services, Inc. (Deer Park, Texas), which was the sixth facility of the series. Facility reports similar to this one have been published for the first five tests which were conducted at the Marquardt liquid injection facility in Van Nuys, California; the Surface Combustion pyrolysis facility in Toledo, Ohio; the Systech fluidized bed reactor in Franklin, Ohio; the Zimpro wet air oxidation unit in Rothschild, Wisconsin; and the 3 M rotary kiln incinerator in Cottage Grove, Minnesota. The facility reports are primarily of an objective nature presenting the equipment description, waste analysis, operational procedures, sampling techniques, analytical methods, emission data and cost information. Facility reports are published as soon as possible after the testing has been completed at a facility so that the raw data and basic results will be available to the public quickly.

In addition to the facility reports, a final report will also be prepared after all testing has been completed. In contrast to the facility reports which are primarily objective, the final report will provide a detailed subjective analysis of each test and the overall program.

## ACKNOWLEDGMENTS

TRW wishes to express its sincere appreciation to Rollins Environmental Services, Inc., particularly Messrs. Donald Matter and Jerry Neel, for their cooperation in conducting these facility tests. The project is also deeply indebted to Mr. Eugene Crumpler of the Office of Solid Waste Management Programs, U.S. Environmental Protection Agency, for his advice and technical direction.

PB 270 897

# DESTROYING CHEMICAL WASTES IN COMMERCIAL-SCALE INCINERATORS

## (Facility Report 6)

*This final report (SW-122c.6) describes work performed*
*for the Federal solid waste management program*
*under contract no. 68-01-2966*
*and is reproduced as received from the contractor.*

U.S. ENVIRONMENTAL PROTECTION AGENCY

1977

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

## CONTENTS

Page

1. Summary 1

2. Introduction 4

3. Process Description 6

3.1 Facility Process 6

3.1.1 Incineration System 6
3.1.2 Waste Feed Systems 6
3.1.3 Auxiliary Fuel Feed Systems 6

3.2 Instrumentation 8

3.3 Emission Control System 8

4. Test Description 9

4.1 TRW Tests of PCB-containing Capacitors 9

4.1.1 Waste Tested 9
4.1.2 Operational Procedures 11
4.1.3 Sampling Methods 15
4.1.4 Analysis Techniques 24
4.1.5 Problems Encountered 30

4.2 ADL Tests of Nitrochlorobenzene Waste 31

4.2.1 Waste Tested 31
4.2.2 Operational Procedures 32
4.2.3 Sampling Methods 33
4.2.4 Analysis Techniques 36
4.2.5 Problems Encountered 37

5. Test Results 39

5.1 Results of PCB Tests 39

5.1.1 Operating Conditions for Tests 39
5.1.2 Composition of Combustion Zone Gas 41
5.1.3 Final Emissions 44

CONTENTS (CONTINUED)

Page

5.2 Results of NCB Tests 58

5.2.1 Operating Conditions for Tests 58

5.2.2 Destruction Efficiency and Composition of Combustion Zone Effluent Gas 58

5.2.3 Final Emissions 66

6. Waste Incineration Cost 69

6.1 Capital Investment 69

6.2 Annual Operating Costs 69

7. References 78

Appendixes

Appendix A - Assessment of Environmental Impact of Destroying Chemical Wastes 79

Appendix B - Sample Volume Data - PCB Tests 82

Appendix C - Analytical Chemistry Details - PCB Tests 86

Appendix D - PCB Analysis by GC/MS 88

Appendix E - Sample Volume Data - NCB Tests 96

Appendix F - Analytical Chemistry Details - NCB Tests 100

Appendix G-1 - Rollins Environmental Services, Inc. Operating Data 123

Appendix G-2 - Incineration System Raw Data 145

Appendix H - Calculation of Waste Destruction Performance 160

## FIGURES

| | | Page |
|---|---|---|
| 3-1 | Schematic of Rollins Environmental Incinerator | 7 |
| 4-1 | Fiber Drums Being Fed to Rotary Kiln | 14 |
| 4-2 | Sampling Sites at Rollins | 17 |
| 4-3 | Sampling System for On-line Instruments | 18 |
| 4-4 | Instrument Racks | 19 |
| 4-5 | Combustion Zone Sampling Train Schematic | 20 |
| 4-6 | Water-cooled Probe Design | 22 |
| 4-7 | Combustion Zone Sampling Site | 23 |
| 4-8 | Stack Sampling Site | 25 |
| 4-9 | Hot Zone Sampling Train for NCB Tests | 36 |
| 5-1 | Filters From Combustion Zone and Stack Gas Sampling Trains | 45 |
| 5-2 | Photograph of Solid Residue Samples | 53 |
| F-1 | Sorbent Trap Extractor | 103 |
| F-2 | GC Calibration Curve for NCB Standards | 106 |

# TABLES

| | | Page |
|---|---|---|
| 1-1 | Results Summary | 2 |
| 4-1 | Composition of PCB Survey Sample | 10 |
| 4-2 | Composition of PCB Representative Sample | 11 |
| 4-3 | Description of On-line Instruments | 21 |
| 4-4 | Summary of Analytical Methods | 29 |
| 5-1 | Incinerator System Parameters Data Summary | 40 |
| 5-2 | Gas Composition Data Summary | 41 |
| 5-3 | Summary of Survey Analysis on the Combined Probe Wash and Particulate Filter Extracts | 43 |
| 5-4 | Summary of Survey Analysis on Sorbent Trap Extracts | 43 |
| 5-5 | Survey for Trace Metals in Combustion Zone Samples by ICPOES | 46 |
| 5-6 | Detection Limits for Elements Not Found in the Combustion Zone Samples by ICPOES | 47 |
| 5-7 | Particulate Loading in the Effluent Gas | 47 |
| 5-8 | Survey for Trace Metals in Stack Samples by ICPOES | 48 |
| 5-9 | Detection Limits for Elements Not Found in the Stack Samples by ICPOES | 49 |
| 5-10 | Summary of Survey Analysis of Scrubber Water Extracts | 50 |
| 5-11 | Survey for Trace Metals in Scrubber Water Samples by ICPOES | 51 |
| 5-12 | Detection Limits for Elements Not Found in the Scrubber Waters by ICPOES | 52 |
| 5-13 | Results of Test III Solid Residue Analysis for PCBs by GC/MS | 54 |
| 5-14 | Summary of Survey Analysis of Solid Residue Extracts | 54 |
| 5-15 | Analysis of Solid Residues by ICPOES | 56 |
| 5-16 | Analysis of Solid Residues by SSMS | 57 |

## TABLES (CONTINUED)

Page

5-17 Operating Conditions for Tests on NCB Wastes 59

5-18 Summary of Quantities of Organic Materials in Hot Zone Effluent Sample Extracts 60

5-19 Hot Zone Emission Rates and Calculated Destruction Efficiencies 61

5-20 Quantitative Analysis Results Obtained from On-line Instruments and Gas-Detecting Tubes 63

5-21 Organic Chemical Species Found in Hot Zone Effluent Sample Extracts 65

5-22 Chloride and Nitrate Analyses of Scrubber Wastes 68

6-1 Capital Investment - 5000 Metric Tons/Year Used PCB Capacitor Waste Incineration Plant 70

6-2 Capital Investment - Central Facility for Incineration 4540 Metric Tons/4-Month Period NCB Waste 72

6-3 Capital Investment - 4540 Metric Tons/Year NCB Waste Incineration Plant (On-Site) 73

6-4 Annual Operating Cost - 5000 Metric Tons/Year Used PCB Capacitor Waste Incineration Plant 75

6-5 Pro-Rata Annual Operating Cost - Central Facility for Incineration - 4540 Metric Tons/4-Month Period NCB Waste 76

6-6 Annual Operating Cost - 4540 Metric Tons/Year NCB Waste Incineration Plant (On-Site) 77

B-1 Sampling System Data Summary 83

B-2 Rollins Sample Gas Volumes at Standard Conditions 84

B-3 Collected Water Volume Data 85

D-1 AMUs to Be Searched for Raw Counts 94

E-1 Stack Sampling Data 97

E-2 Hot Zone Sampling Data 98

E-3 Estimated Total Gas Effluent Flow Ratio 99

## TABLES (CONTINUED)

Page

F-1 Volumes of Impinger Solutions 109

F-2 Gravimetric Data for Probe Wash, Filter, and Dry Impinger Samples 110

F-3 Results of Gravimetric Analyses on Concentrated Organic Extracts 111

G-1 Test I - TRW Background 143

G-2 Test II - Hammermilled PCB Capacitors 146

G-3 Test III - Whole PCB Capacitors 148

G-4 Run R1(B) Background Test 150

G-5 Run R2 - NCB to Kiln 153

G-6 Run R3 - NCB - Diesel Fuel to Loddby 156

G-7 Run R4 - NCB - Diesel Fuel to Loddby 158

# 1. SUMMARY

Incineration tests of selected chemical wastes were conducted at a contract disposal facility operated by Rollins Environmental Services, Inc., in Deer Park, Texas. This incineration system, consisting of a rotary kiln and a liquid injection burner feeding a common afterburner, is representative of commercial waste destruction equipment presently in use. Total heat release of the system was 28 million kcal/hr (110 million Btu/hr). These tests were performed to determine the effectiveness of thermal destruction of two different industrial wastes: 1) discarded electrical capacitors containing polychlorinated biphenyls (PCBs), and 2) waste from production of nitrochlorobenzene (NCB).

The PCB-containing capacitors were incinerated in the rotary kiln both as a hammermilled "fluff" and as whole capacitors. PCB tests were performed at maximum incinerator temperatures and residence time to operate under conditions of maximum destructive effectiveness. NCB waste was destroyed in the liquid injection burner at two different waste feed conditions to determine the effects of operating variables. Number 2 oil was utilized as auxiliary fuel in the destruction of each waste. A background test with auxiliary fuel only was performed prior to initiation of the PCB tests and again just before the NCB burns to obtain baseline combustion and emission data.

During each test, combustion products were continuously monitored by on-line instrumentation. Samples were taken of the combustion zone effluent to evaluate the destruction effectiveness. Stack gases and waste products (scrubber water, ash, etc.) were collected to verify the environmental safety of the tests. The PCB tests were monitored by a TRW sampling team, while the NCB test samples were taken by a team from Arthur D. Little, Inc., under subcontract with TRW. Laboratory analyses of test samples were performed by each organization for their respective waste tests.

Results of the waste destruction tests are summarized in Table 1-1. It should be noted that particulate stack emissions were higher for the whole capacitor test (53 mg/$m^3$) compared to the hammermilled capacitor test (35 mg/m3). Of significance also is the fact that while emissions of HCl from the incinerator alone were high (circa 6 g/$m^3$), the wet scrubber removed 99.8 percent of the HCl from the effluent and resulting stack emissions were approximately 13 mg/$m^3$.

No waste constituents were found in any of the combustion gas samples above the minimum detection limits indicated in Table 1-1. Incineration of each waste was accomplished with high effectiveness, with overall waste destruction efficiencies of over 99.999 percent for every test except incineration of whole capacitors, where waste residuals in the ash reduced the overall destruction efficiency to approximately 99.5 percent. No PCB concentrations above 0.1 mg/kg were found in the ash from destruction of

# TABLE 1-1. RESULTS SUMMARY

| | PCB-Containing Capacitors | | Nitrochlorobenzene Waste | |
|---|---|---|---|---|
| | Hammermilled | Whole | 1st Test | 2nd Test |
| Kiln Flame Temperature (°C) | 1252 | 1330 | Not Used | Not Used |
| Afterburner Temperature (°C) | 1331 | 1332 | 1307 | 1337 |
| Calculated Residence Time (sec) | 3.2 | 3.0 | 2.3 | 2.1 |
| Solid Waste Feed (kg/hr) | 210 | 360 | None | None |
| Liquid Waste Feed (l/hr) | None | None | 404 | 353 |
| Number 2 Oil Feed (l/hr) | 2411 | 2300 | 1619 | 1410 |
| Quality of Stack Emission: | | | | |
| Particulate (mg/m$^3$) | 35 | 53 | 14 | 16 |
| Hydrochloric Acid (mg/m$^3$) | Not Analyzed | Not Analyzed | 13 | 13 |
| Trace Metals (mg/m$^3$) | 2.7 Pb | 3.3 Pb | <0.01Ti,Ni,Cr[a] | <0.01Ti,Ni,Cr |
| Quality of Combustion Gas: | | | | |
| Total Organics (mg/m$^3$) | 14 | 23 | 42 | 53 |
| Waste Content (mg/m$^3$) | Not Detected (<0.005) | Not Detected (<0.005) | Not Detected (<0.05) | Not Detected (<0.05) |
| Trace Metals (mg/m$^3$) | 12Pb,1.0Zn | 11Pb,2.6Zn | <0.01Ti,Ni,Cr | <0.01Ti,Ni,Cr |
| Quality of Scrubber Water: | | | | |
| Total Orangics (mg/l) | 0.19 | 0.73 | 1.0 | 1.4 |
| Trace Metals (mg/l) | 1.7Pb, 1.5Zn | 4.0Pb,1.3Zn | <0.1Ti,Ni,Cr | <0.1Ti, Ni,Cr |
| Chloride Ion (mg/l) | Not Analyzed | Not Analyzed | 1725 | 1815 |
| pH | 5 | 5 | 3 | 3 |
| Quality of Ash: | | | | |
| Waste Content (mg/kg ash) | Not Detected (<0.1) | 470 | No Ash | No Ash |
| Destruction Efficiency: | | | | |
| Total Organics (percent) | 99.98 | 99.96 | 99.94 | 99.87 |
| Waste Constituents (percent) | >99.999 | 99.5[b] | >99.999 | >99.999 |

(a) Trace metal contents for NCB effluents were calculated from spark source mass spectrometric analysis of representative waste sample.

(b) Overall destruction efficiency reduced due to high waste content in ash. Efficiency based on combustion gas sampling was >99.999 percent.

hammermilled capacitors. Total organic destruction efficiencies ranged from 99.04 to 99.98 percent.* In general, the capability of the Rollins incineration system to destroy either solid or liquid wastes was effectively demonstrated during these tests.

Capital and operating cost estimates were made for incinerator systems similar to the Rollins unit to destruct each of the two wastes. Estimated capital investment, not including land cost, for a facility to hammermill and incinerate 5000 metric tons of waste capacitors per year is $3.65 million, with the total fixed and variable operating costs equivalent to $741 per metric ton of waste destroyed. Over $300 per ton of operating cost can be saved if a high heat content waste is utilized instead of Number 2 fuel oil to provide temperatures required to destroy the PCB. Using a waste to incinerate the capacitors would be the actual commercial destruction method; however, in order to more precisely analyze the PCB combustion products, a clean-burning fuel oil was used as auxiliary fuel for these tests.

Estimated operating cost for destruction of 4540 metric tons per year of NCB waste (one half of estimated total volume generated annually) in a central, contractor-owned facility such as Rollins is $242 per metric ton, including cost of transporting wastes. For this central facility, presumed to operate with NCB waste over a four-month period of each year, the estimated capital investment is $3.75 million; therefore, the pro-rata capital investment for NCB destruction would be $1.25 million (one-third use of facility). For an on-site facility, scaled to a size adequate for destruction of 4540 metric tons per year of NCB waste only, the estimated capital investment is $2.82 million and the estimated operating cost is $283 per metric ton.

---

*Destruction efficiency for total organics compares the input rate of combined waste and auxiliary fuel to the emitted rate of all organic material found in the combustion samples. Waste destruction efficiency compares only waste input rate to concentration of organic waste constituents in the combustion gas. All combustion zone samples were taken in the afterburner exit duct prior to the scrubber system. A sample destruction efficiency calculation is presented in Appendix H.

## 2. INTRODUCTION

The U.S. Environmental Protection Agency has sponsored a program* to evaluate the effectiveness of a variety of types of commercial thermal destruction facilities for destroying chemical wastes. This report describes test operations and results of incinerating two different wastes, PCB-containing capacitors and nitrochlorobenzene (NCB), at a contract disposal facility operated by Rollins Environmental Services, Inc., in Deer Park, Texas.

The Rollins incineration system, consisting of a rotary kiln and a liquid injection burner feeding a common afterburner, was selected as representative of full-scale waste destruction equipment presently in commercial use. Rotary kiln incineration represents a well-established technology with widespread applicability to a variety of hazardous wastes. Total heat release is 28 million kcal/hr, with combustion gas temperatures of 1300°C in the afterburner. Stack emissions are controlled by a venturi scrubber system.

The two wastes tested at the Rollins facility, PCB-containing capacitors and nitrochlorobenzene production waste, were selected on the basis of their suitability for destruction in a rotary kiln incinerator. A conveyor system was used to feed either hammermilled or whole capacitors into the kiln. The afterburner provided additional residence time at temperatures sufficient to insure efficient destruction of the PCB in the capacitors. NCB waste was selected to be burned in the kiln, but did not atomize properly by itself in the kiln burner nozzle at Rollins. After being mixed with No. 2 oil, the NCB was fed through the liquid injection burner which fired the afterburner.

Polychlorinated biphenyls (PCBs) have been used extensively in capacitor dielectric fluids, with an estimated 11,000 metric tons per year produced for this use. Although other materials are now being used as substitutes for PCB, thousands of tons of capacitors, each containing 20 to 40 percent PCB by weight, are taken out of service annually due to failure or obsolescence. PCB is very resistant to chemical and biological degradation; therefore, incineration or thermal degradation is the most feasible means of destruction. Because of the extreme stability of PCB, sufficient residence time at high temperature is essential for destruction.

The principal use of nitrochlorobenzene is as an intermediate material in the manufacture of synthetic dyes, drugs, pesticides, and photochemicals. Estimated annual production of NCB waste similar to that tested is 9,100 metric tons.

---

*Contract No. 68-01-2966

The following report sections describe in detail the incinerator process equipment (Section 3), and the wastes destroyed and test and sampling procedures followed (Section 4). Test results are presented and discussed (Section 5), including effectiveness of destruction of the wastes. An estimate of the capital investment and operating costs of disposing of wastes using this type of incinerator equipment is also included in the report (Section 6).

# 3. PROCESS DESCRIPTION

## 3.1 FACILITY PROCESS

The Rollins incineration facility process is shown schematically in Figure 3-1. Basic system components include:

- Rotary kiln incinerator
- Waste liquid burner
- Afterburner
- Waste feed systems
- Auxiliary fuel feed systems
- Instrumentation
- Emission control system

Following is a description of the incineration and feed systems. Facility instrumentation and emission controls are discussed in subsequent Sections 3.2 and 3.3, respectively.

### 3.1.1 Incineration System

The incineration system consists of a rotary kiln and a liquid injection burner, both feeding a common afterburner (Figure 3-1). Total heat release is 28 million kcal/hr. The kiln is 4.9 meters long and 3.2 meters in diameter. Flame temperature in the kiln is nominally 1300°C.

The waste liquid burner (Loddby) measures 4.9 meters in length by 1.6 meters diameter. Loddby flame temperature is approximately 1500°C. Resulting afterburner gas temperature is typically 1300°C. Afterburner dimensions are: 10.6 meters overall length, 4.0 meters high, and 4.3 meters wide. Overall retention time of the incineration system is from two to three seconds.

### 3.1.2 Waste Feed Systems

Solid wastes, usually packed in fiber drums, are fed into the rotary kiln by a conveyor. Liquids and sludges may also be pumped into the kiln. Liquid wastes that are to be burned in the Loddby can be fed directly from tank trucks or from blending tanks.

### 3.1.3 Auxiliary Fuel Feed Systems

Both the kiln and the Loddby are equipped with natural gas ignitors and gas burners for initial refractory heat-up, flame stability, and supplemental heat, if necessary. Number 2 fuel oil was also used as auxiliary fuel for these tests. Fuel oil was used to provide heat for the PCB tests, since the capacitors by themselves did not have sufficient heat capacity.

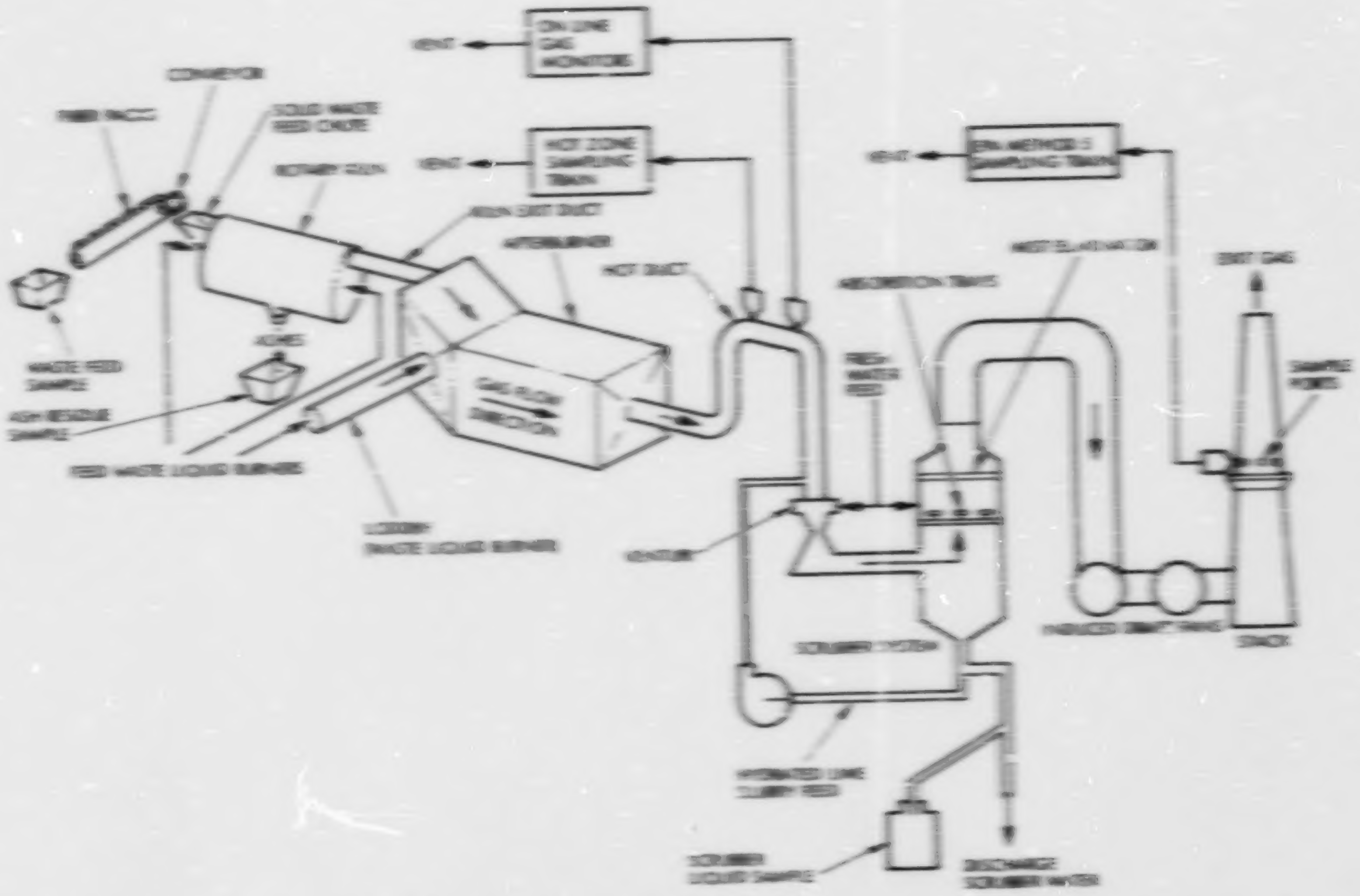

VENT
ON LINE GAS MONITORS
VENT
HOT ZONE SAMPLING TRAIN
CONVEYOR
FIBER PACKS
SOLID WASTE FEED CHUTE
ROTARY KILN
KILN EXIT DUCT
AFTERBURNER
HOT DUCT
WASTE FEED SAMPLE
ASHES
ASH RESIDUE SAMPLE
GAS FLOW DIRECTION
FEED WASTE LIQUID BURNERS
WASTE LIQUID BURNER
VENT
EPA METHOD 5 SAMPLING TRAIN
MIST ELIMINATOR
ABSORPTION TRAYS
EXIT GAS
FRESH WATER FEED
SAMPLE PORTS
VENTURI
INDUCED DRAFT FANS
STACK
SCRUBBER SYSTEM
HYDRATED LIME SLURRY FEED
SCRUBBER LIQUID SAMPLE
DISCHARGE SCRUBBER WATER

Figure 3-1. Schematic of Rollins Environmental Services Incinerator.

The nitrochlorobenzene waste was mixed with fuel oil in order to be fed into the Loddby for incineration.

## 3.2 INSTRUMENTATION

Measurements were made of all process parameters, including temperatures, pressures, and flow rates. On-line gas analysis instruments were also monitored and recorded in the sampling trailer. The following operating temperatures (see Figure 3.1) were recorded during each test: 1) kiln flame, 2) kiln exit duct, 3) Loddby flame, 4) afterburner, and 5) hot duct.

Measured flow rates included: 1) natural gas, 2) auxiliary fuel oil, and 3) waste feed. In addition, scrubber venturi pressure drop and lime consumption were also monitored during tests.

## 3.3 EMISSION CONTROL SYSTEM

Atmospheric emissions from the combustion of solid and liquid wastes during the Rollins incineration tests were controlled by a venturi scrubber, as shown in Figure 3-1. Lime is injected to neutralize the scrubber water. Used scrubber water enters settling ponds where it is analyzed and further treated, if necessary, before discharge.

Exhaust gases also pass through absorption trays and a mist eliminator before entering the stack. The exhaust stack is 30 meters high with standard sampling ports and a platform about 16 meters above ground level.

# 4. TEST DESCRIPTION

This section describes the manner in which the tests were conducted. It is divided into the following subsections, listed in order of discussion:

- Physical and chemical description of the wastes that were tested.
- Operational procedures used and a test-by-test commentary.
- Sampling methods.
- Analysis techniques.
- Description of the problems encountered related to the facility and sampling.

These topics will be discussed separately for the TRW and the ADL portions of the tests conducted at Rollins. TRW first tested PCB-containing capacitor materials; then nitrochlorobenzene (NCB) waste was tested by ADL.

## 4.1 TRW TESTS OF PCB-CONTAINING CAPACITORS

The PCB-containing capacitors were tested both as a hammermilled "fluff" and as whole capacitors. Test burns of these materials were conducted using the rotary kiln incineration system at Rollins, and samples were taken for analysis of the combustion gases, stack gases, scrubber waters, and solid residues of the wastes remaining after incineration.

### 4.1.1 Waste Tested

A survey sample of assorted whole capacitors was received early last year, and a representative sample was later taken during the hammermilling of the capacitors. These waste samples were analyzed for both organic and inorganic composition, and the results are reported in this section.

The survey sample consisted of several types of small capacitors containing PCB fluid. Because each capacitor was different and because it was uncertain whether they represented what would actually be burned in the field tests, it was deemed impractical and unproductive to characterize the whole capacitors (i.e., percent aluminum shell, percent bakelite, percent paper packing, etc.). As a result, analyses were performed only on the fluid obtained by puncturing and draining several of the survey sample capacitors.

The drained PCB fluid was colorless and had a specific gravity of 1.4 at 15.6°C (60°F). The fluid would not ignite in a calorimeter, thus its heating value is estimated to be less than 3,000 kcal/kg (5,000 Btu/lb).

Quantitative elemental analyses for C, H, N, S, and total halogens (as Cl) gave the following results:

| Element | Composition, % |
|---|---|
| C | 54.80 |
| H | 2.63 |
| N | 0.008 |
| S | 0.019 |
| Cl | 42.59 |

Based on the elemental composition, the capacitor fluid approximates Aroclor 1242. The infrared spectrum of the capacitor fluid waste, measured as a thin film between NaCl windows, was also consistent with that of a mixture of polychlorinated biphenyl compounds.

Analysis for trace elements by X-ray fluorescence found Si and P at >500 ppm and 50-500 ppm, respectively, along with chlorine and bromine.

The survey sample of PCB capacitor fluid was chromatographed on a fifty meter SE 30 capillary column which was programmed from 50°C to 250°C at 10°C per minute. The total ion monitor of the mass spectrometer was used as the detector. Chlorinated biphenyl compounds from the monochloro- ($C_{12}H_9Cl$) up through pentachloro biphenyl $C_{12}H_5Cl_5$ were seen. A search specifically for higher molecular weight compounds was conducted, but none were seen. Most, if not all, of the isomers were detected, but it was considered unnecessary to identify each individual isomer. The presence of the nonchlorinated biphenyl species was also noted. The compounds found in the GC/MS of the survey sample are presented in Table 4-1. The estimated concentrations are based on the mass spec total ion monitor and may be somewhat different from normalized percent concentrations from other GC detectors, but the data serve the purpose of showing the distribution of the PCB compounds in the survey sample.

TABLE 4-1. COMPOSITION OF PCB SURVEY SAMPLE

| Compound | Estimated Concentration, (percent w/w) |
|---|---|
| Biphenyl | 0.2 |
| Monochlorobiphenyl (2 isomers) | 2 |
| Dichlorobiphenyl (3 isomers) | 18 |
| Trichlorobiphenyl (6 isomers) | 41 |
| Tetrachlorobiphenyl (13 isomers) | 37 |
| Pentachlorobiphenyl (8 isomers) | 2 |

Only two types of capacitors were selected for the test burns. A portion of these were hammermilled, and a portion were kept to be burned whole. Since the hammermilling process helps to mix and composite the material, a sample of the hammermilled "fluff" was taken to be representative of the feed for both the fluff and the whole capacitor test.

A 20 g portion of the representative (fluff) sample was extracted twice with pentane in a Soxhlet for 24 hours for a total of 48 hours of extraction. The extract was then made to a standard volume of 500 ml. An aliquot of this extract was evaporated to remove all solvent and then weighed. The weight of organic residue represented 29 percent of the extracted fluff sample. To determine nonextractable organics (e.g., paper and plastics), the solid material remaining in the Soxhlet thimble after the pentane extraction was ashed at 800°C for one hour. The remaining ash was 47 percent by weight of the solids sample or 32 percent of the initial 20 g fluff sample.

The pentane extract of the representative sample was analyzed by GC/MS to quantify the types of PCB compounds present. These results are presented in Table 4-2. The composition of the fluff sample is slightly different from that of the survey sample (Table 4-1) but still shows a predominance of the di-, tri-, and tetra-PCB classes. Detection limits for the classes of PCBs (7 to 10 chlorine atoms/molecule) not found in the fluff extract were 0.03 g/kg fluff.

TABLE 4-2. COMPOSITION OF PCB REPRESENTATIVE SAMPLE

| Compound | Estimated Concentration | |
|---|---|---|
| | g PCB/kg fluff | Percent of Total PCB Compounds |
| Monochlorobiphenyl | 1.0 | 0.2 |
| Dichlorobiphenyl | 94 | 20 |
| Trichlorobiphenyl | 150 | 31 |
| Tetrachlorobiphenyl | 180 | 37 |
| Pentachlorobiphenyl | 55 | 11 |
| Hexachlorobiphenyl | 3.3 | 0.7 |

### 4.1.2 Operational Procedures

Detailed operating procedures, including both a test plan and a safety plan, were reviewed and approved prior to the arrival of the TRW sampling team on site. Procedures and operating conditions were also recorded during the field tests. Following are brief summaries of both plans, a test-by-test commentary on events that occurred in the field, and information on the disposal of the waste residues.

#### 4.1.2.1 Test Procedures

The PCB tests conducted at Rollins consisted of one test burn of waste capacitors that had been hammermilled and sealed in 130 liter (35 gallon) fiberpacks and one test burn of whole waste capacitors. The basic procedures for each test are listed below:

- Verify instrumentation and sampling systems ready.
- Connect auxiliary fuel (No. 2 oil) lines to rotary kiln and Loddby.
- Obtain a sample of the fresh scrubber water.
- Switch the kiln and Loddby to fuel oil, stabilize temperatures, and purge for 1 to 2 hours.
- Activate on-line combustion gas analyzer system.
- Initiate waste feed to rotary kiln.
- Stabilize flows and temperatures.
- Extended burn duration:
    - Process data acquisition.
    - Combustion gas composition data acquisition.
    - Combustion zone and stack gas sampling.
    - Scrubber liquid sampling.
- Collect samples of residue from the rotary kiln.
- Switch the kiln and Loddby burners back to Rollins waste materials.

The two tests consisted of burn periods during which a 3-hour combustion gas sample was to be acquired at steady-state operating conditions. A 3-hour sampling run while burning with No. 2 oil only was also required to obtain background data. As explained in the test commentary (Section 4.1.2.3), it was not always possible to obtain a 3-hour sample. Stack gases were to be sampled by the standard EPA Method 5 procedure.

Because of the limited amount of available waste material, Rollins was asked to use their operating experience and set their optimum operating conditions for the tests. Target temperatures were as follows:

| | |
|---|---|
| Afterburner | 1260 to 1320°C |
| Hot duct | 1090°C |
| Loddby flame | 1480°C |
| Kiln flame | 1320°C |

#### 4.1.2.2 Safety Procedures

Safety requirements established and maintained for handling and incinerating these specific wastes included the following:

- Only authorized personnel with prior approval were permited in the test area during operations.
- Waste handling was performed only by personnel wearing suitable protective clothing and trained in handling such materials.
- Visual observation of the test system was maintained at all times during operation.
- Canister gas masks and emergency oxygen resuscitation units were available in the immediate area.
- Telephone numbers of emergency agencies were posted near the test area.

#### 4.1.2.3 Test Commentary

Two test burns of PCB-containing capacitors were conducted at Rollins, one using whole capacitors and one using a "fluff" obtained by hammermilling. Since auxiliary fuel was required for the combustion of both forms of capacitors, a baseline test was also conducted with No. 2 oil.

Test I - No. 2 Oil Background Test

A baseline test was performed with No. 2 oil to acquire background data on the performance of the rotary kiln system while burning only auxiliary fuel. A 2.0-hour combustion zone sample was procured while the system was operating at steady-state conditions of 1306°C kiln and 1308°C afterburner average temperatures. No stack sample was obtained due to severe storm conditions at the time. This problem is discussed further in Section 4.1.5. Because of the resulting lack of stack velocity data, residence time was not calculated for this test. Scrubber water samples were secured. There was no solid residue from the kiln generated during this test.

Test II - PCB "Fluff" Test

The hammermilled fluff of PCB-containing capacitors had been packaged for storage in polyethylene-lined fiber drums. These drums were fed whole via the conveyor belt into the rotary kiln as shown in Figure 4-1. The kiln was first purged with a 1.5-hour burn of No. 2 oil alone before beginning testing of the PCB fluff. As the first fiber drum was fed, the on-line monitors were watched for any sign of combustion problems. The kiln maintained essentially steady-state conditions, and no surge of unburned hydrocarbons was observed. Rollins then proceeded to feed drums at a rate of one every four or five minutes, and sampling was begun. The fiber drums burned in this test weighed an average of 25 kg with an approximate tare weight of 9 kg, indicating that roughly 210 kg per hour of PCB fluff was fed into the rotary kiln. Temperatures averaged 1252°C in the kiln and 1321°C in the afterburner. Residence time was calculated to be 3.2 seconds.

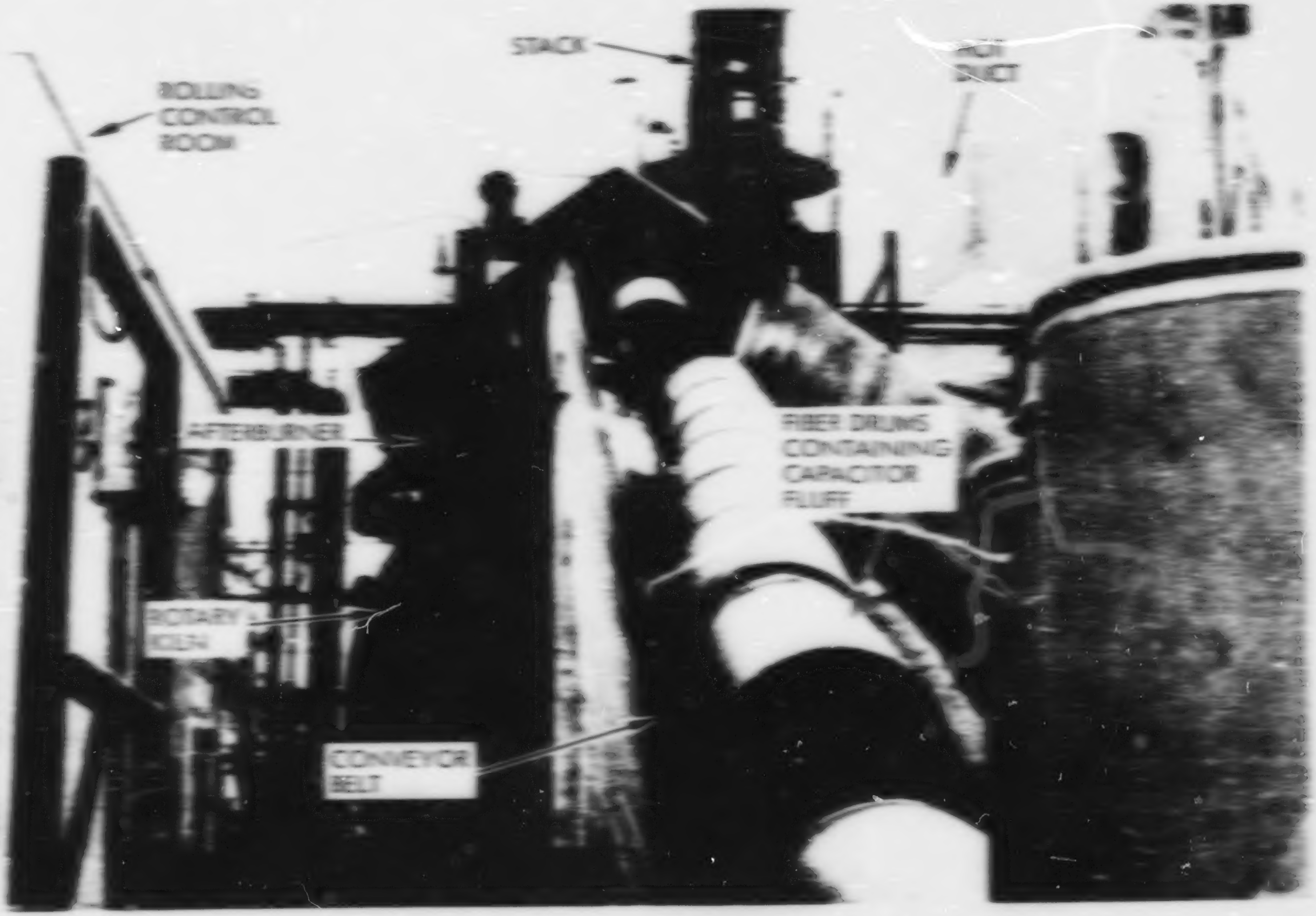

ROLLING CONTROL ROOM
STACK
HOT DUCT
AFTERBURNER
FIBER DRUMS CONTAINING CAPACITOR FLUFF
ROTARY KILN
CONVEYOR BELT

Figure 4-1. Fiber drums being fed to rotary kiln.

The sampling train at the combustion zone collected sample for 2.25 hours before a power failure at the sampling site forced the train to be shut down. A full one-hour stack sample was obtained. Scrubber water samples were acquired and 2 kg of the solid residue from the test was taken as a sample.

After completion of the sampling efforts, the Rollins facility changed from No. 2 oil to their normal alkyl waste fuel and continued to feed the few remaining fiber drums of the PCB fluff while watching the on-line gas monitors. Composition of the gas leaving the afterburner remained the same while burning with the alkyl fuel, and no increase in unburned hydrocarbons was observed.

Test III - Whole PCB-Containing Capacitors

After a 1.25-hour purge on No. 2 oil, the first whole capacitors were fed. The whole capacitors were removed from the steel drums they had been shipped in and were fed into the rotary kiln individually via the conveyor belt. The initial feed rate proved too fast as 20 minutes into the capacitor burning, an increase of 20 to 30 ppm of unburned hydrocarbons passing the afterburner was observed on the on-line hydrocarbon monitor. The feed rate was subsequently reduced to approximately two drums of capacitors per hour. A total of six drums was burned at an average weight of 205 kg each. Tare weight of the drums was approximately 24 kg, yielding an average feed rate of 360 kg of capacitors per hour.

Steady conditions were then maintained throughout the remainder of the test except for a 15-minute period during which no capacitors were fed because of a conveyor belt malfunction. Temperatures averaged 1339°C in the kiln and 1332°C in the afterburner, with a calculated residence time of 3.0 seconds.

After 2.25 hours of sampling, the combustion zone train was shut down when Rollins ran out of capacitors to feed. A full one-hour stack sample was obtained along with scrubber water and solid residue samples.

#### 4.1.2.4 Disposal of Waste Residues

All of the PCB capacitor material consigned by Rollins for these tests was consumed in the tests. The steel drums that had contained the whole capacitors were landfilled in an approved Class I site. The scrubber waters were piped to treatment ponds at the Rollins site, and the solid residues were trucked from the kiln's hopper to an approved Class I site and landfilled.

### 4.1.3 Sampling Methods

Sampling methods used in the tests at Rollins were chosen to cover three basic areas:

1) Continuous, on-line monitoring of gas composition to determine and follow steady state conditions.

2) Collection and concentration of hot zone combustion products to identify and quantify the trace organic and inorganic species formed.

3) Collection of final emission and waste products to evaluate the environmental safety of the tests.

Following is a brief summary of the methods for each of these areas. More detailed discussions can be found in the Rollins Analytical Plan (Reference 1). The locations of the trailer and sampling trains at the site are shown in Figure 4-2.

### 4.1.3.1 On-Line Gas Monitoring

Gases were drawn continuously from the hot zone just downstream of the afterburner through a ceramic probe, and then through a heated Teflon sample line to the trailer. The gas then entered the system shown in Figure 4-3. The gas conditioner supplied a cool, dry, particulate-free sample to all of the analyzers except the hydrocarbon (HC) monitor which used an untreated sample. A heated Teflon line carried the HC gas sample from a tee in the unconditioned sample line to the HC analyzer.

The monitoring instruments used are listed with their operating ranges in Table 4-3. Data was recorded on Hewlett-Packard 680M strip chart recorders. Figure 4-4 shows the instrument racks mounted in the sampling trailer. The analyzers, recorders, and manifold valves were all located in racks to provide ease of operation and accessibility.

### 4.1.3.2 Combustion Zone Sample

The sampling train used to collect hot zone gases, vapors, and particulate matter is shown schematically in Figure 4-5. It consists of a standard EPA Method 5 train with the following important modifications presented in order according to flow direction through the train:

- There was a stainless steel jacketed, water-cooled probe (shown schematically in Figure 4-6) with a quartz liner. The liner provides an inert surface for the sample gas, and the cooled, stainless steel jacket cools this gas in order to quench any further reactions of the sample constituents and to yield a gas temperature compatible with train materials. Further cooling of the gas was achieved by aspirating an air/water mixture into the space between the steel jacket and quartz liner.

- A chromel/alumel thermocouple was potted into the dogleg going from the quartz probe liner to the filter housing to check the temperature of the gas stream at that point.

- An ultra high-purity glass fiber filter was used, Gelman Spectro-quality Type A. The filters, which were muffled to remove organics, have extremely low background levels of inorganics. They were

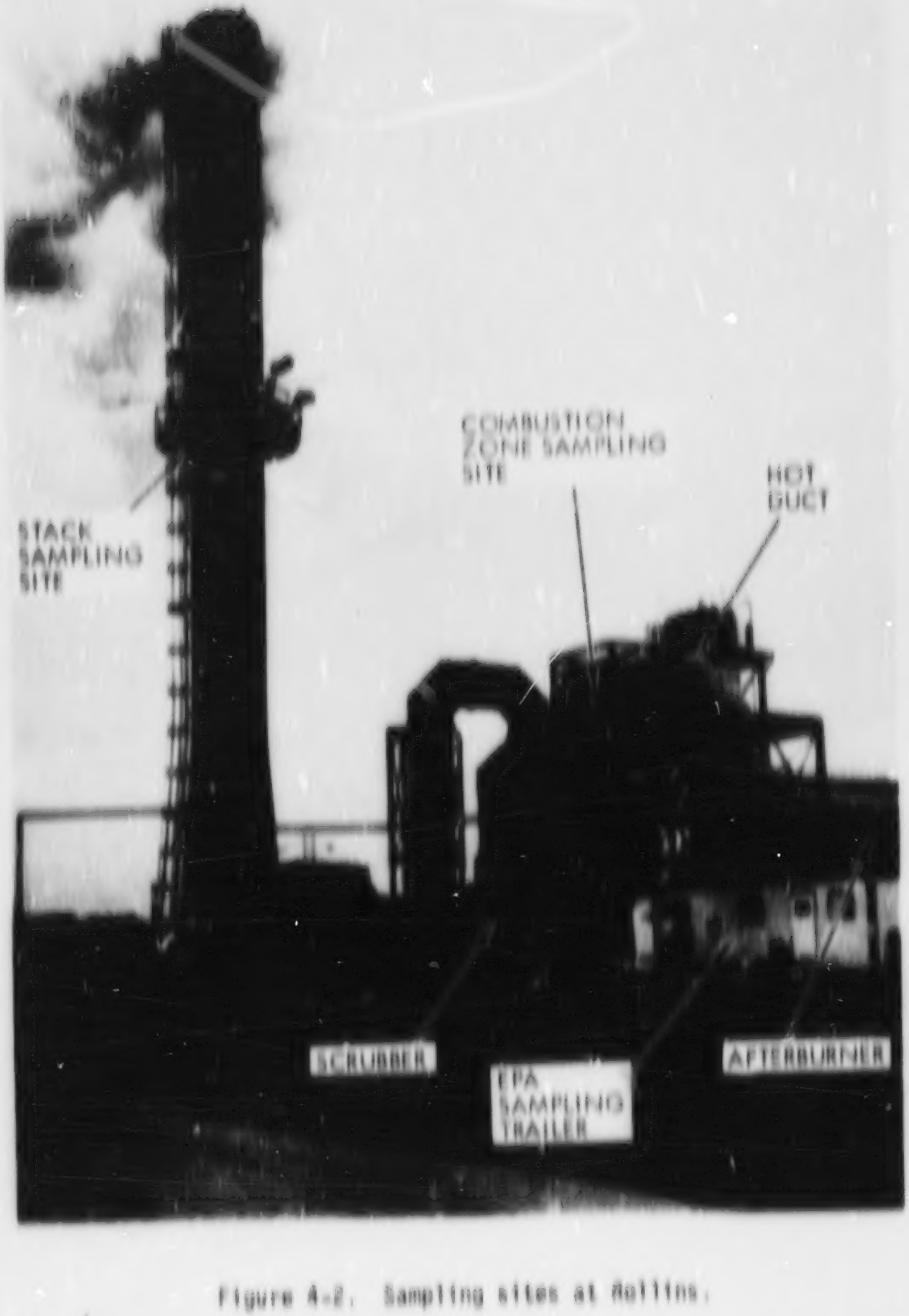

STACK SAMPLING SITE
COMBUSTION ZONE SAMPLING SITE
HOT DUCT
SCRUBBER
EPA SAMPLING TRAILER
AFTERBURNER

Figure 4-2. Sampling sites at Rollins.

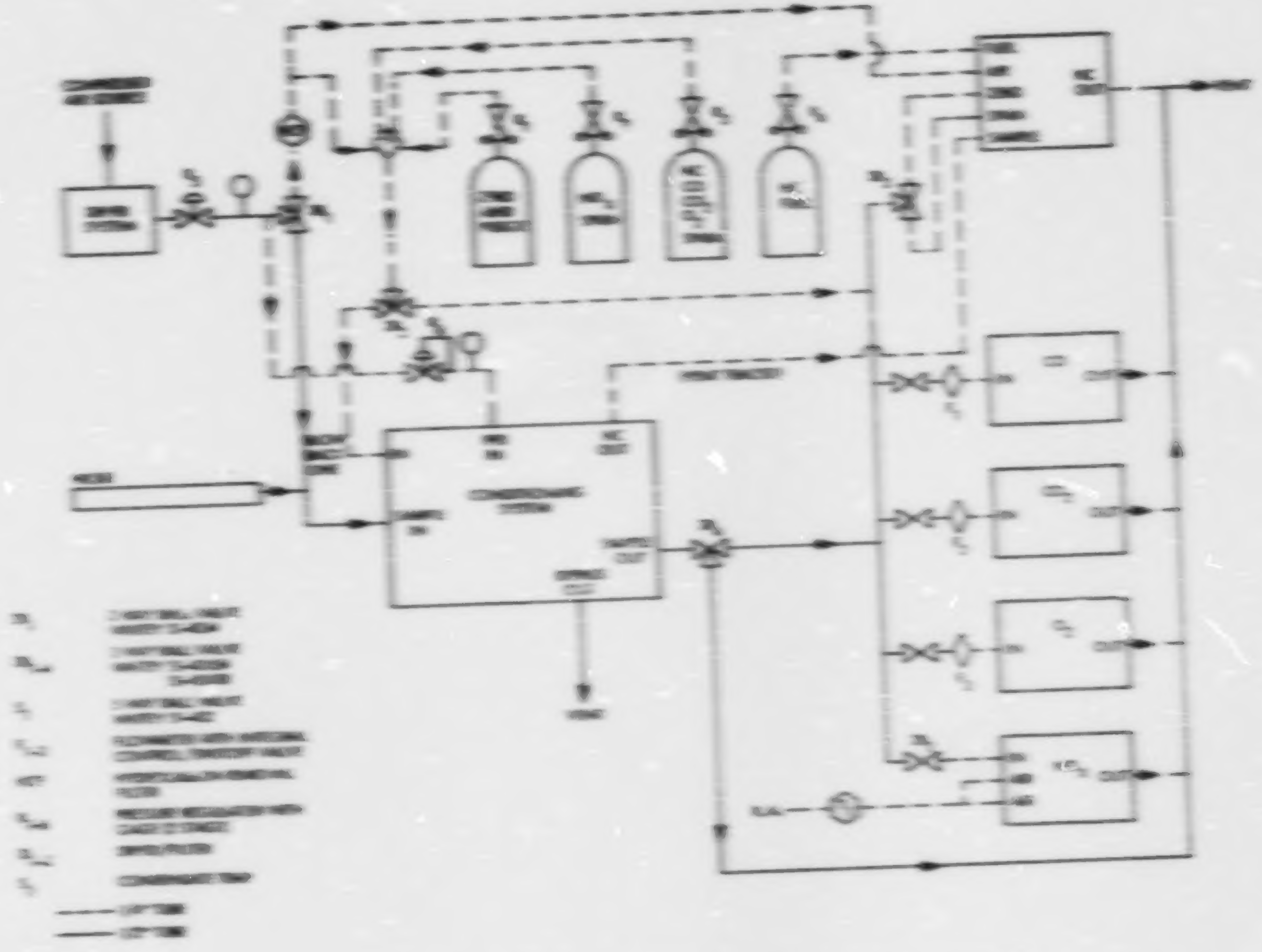


Figure 4-1. Sampling system for on-line instruments.

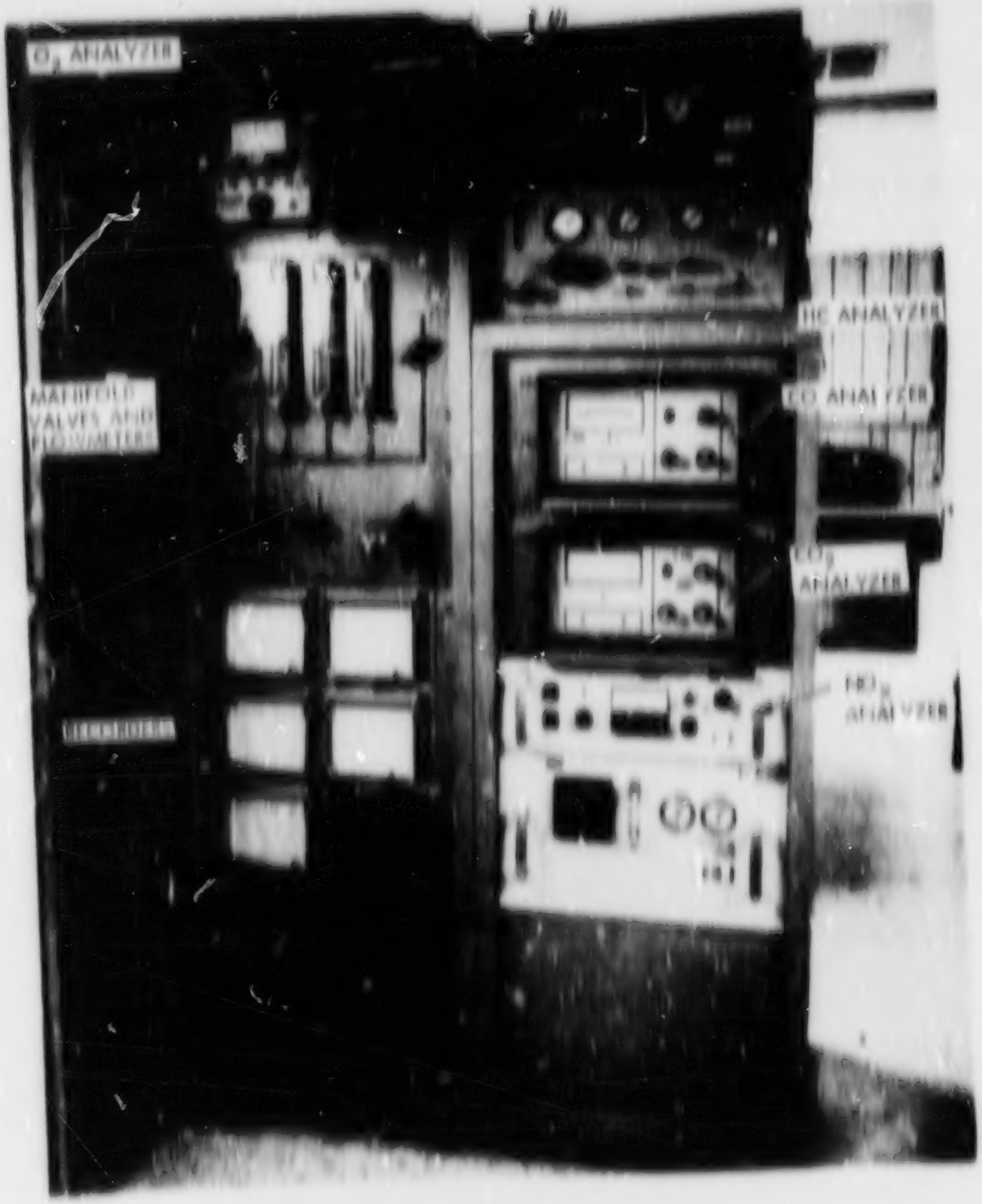

$O_2$ ANALYZER
MANIFOLD VALVES AND FLOWMETERS
RECORDERS
HC ANALYZER
CO ANALYZER
$CO_2$ ANALYZER
$NO_x$ ANALYZER

Figure 4-4. Instrument racks.

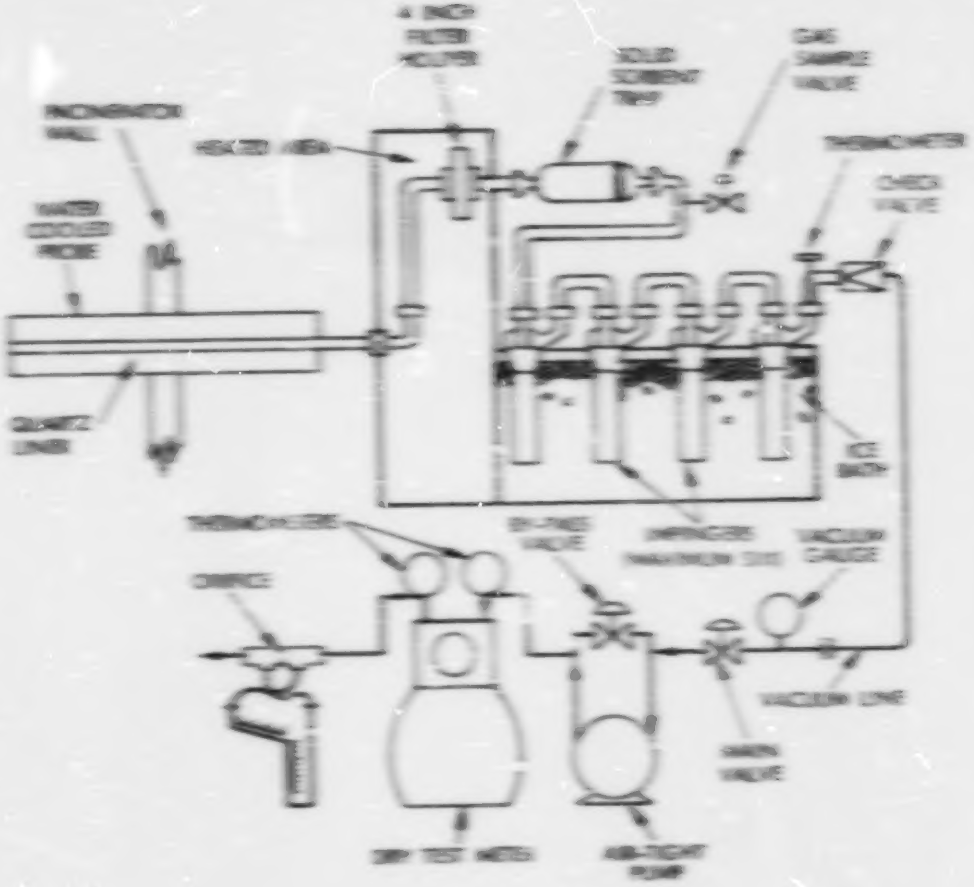

4 INCH FILTER HOLDER
SOLID SORBENT TRAP
GAS SAMPLE VALVE
INCINERATOR WALL
HEATED AREA
THERMOMETER
CHECK VALVE
WATER COOLED PROBE
QUARTZ LINER
ICE BATH
THERMOMETERS
BY-PASS VALVE
IMPINGERS
VACUUM GAUGE
ORIFICE
VACUUM LINE
MAIN VALVE
DRY TEST METER
AIR-TIGHT PUMP

Figure 4-5. Combustion zone sampling train schematic.

tared by desiccating and weighing on consecutive days to a constant weight (±0.1 mg), then stored and handled throughout the tests and analyses in glass petri dishes.

- A solid sorbent trap, designed to absorb the organic constituents in the sample gas stream, is located downstream of the heated filter and upstream of the first impinger. The sorbent trap, with overall dimensions of 170 × 45 mm, contained ∿40 g of XAD-2, an Amberlite resin of the type commonly used as a chromatographic support.

TABLE 4-3. DESCRIPTION OF ON-LINE INSTRUMENTS

| Species Analyzed | Manufacturer and Model | Range* |
|---|---|---|
| Total hydrocarbons (HC) | Beckman Model 402 | 0.05 ppm – 10% with eight ranges |
| Carbon monoxide (CO) | Beckman Model 865 | 2-200 ppm<br>10-100 ppm |
| Carbon dioxide ($CO_2$) | Beckman Model 864 | 0.05 – 5%<br>0.02 – 20% |
| Oxygen ($O_2$) | Taylor OA 273 | 0.05 – 5%<br>0.25 – 25%<br>1 – 100% |
| Sulfur Dioxide ($SO_2$) | Envirometrics | 0 – 100 ppm |
| Oxides of nitrogen ($NO_x$) | Thermo Electron Model 10-A | 0.05 – 10,000 ppm with eight ranges |

*All of these manufacturers report an accuracy of ±1 percent of full scale for their instruments.

This hot zone train was operated at a flow rate of approximately 30 liters/min for two hours during each test, thereby sampling an average of 3 to 4 cubic meters. Gas volumes were measured to 0.03 liter, with a leak rate of less than 0.6 liter/min. Operating parameters for the train and sample volume data are tabulated in Appendix B. The location of the hot zone sampling train at the test site is shown in Figure 4-7.

The following hot zone samples were obtained from the sampling train of each test:

- Solvent probe wash.

- Aqueous probe wash.

- 10-cm diameter particulate filter.
- Solid sorbent trap.
- Combined impinger solutions.
- Acidified split of combined liquid impingers.
- Solvent wash of impinger glassware.
- Spent silica gel.

In addition to the samples from the hot zone train, portions of the combustion gas were collected in Tedlar bags from the sample bypass port of the on-line hydrocarbon analyzer.

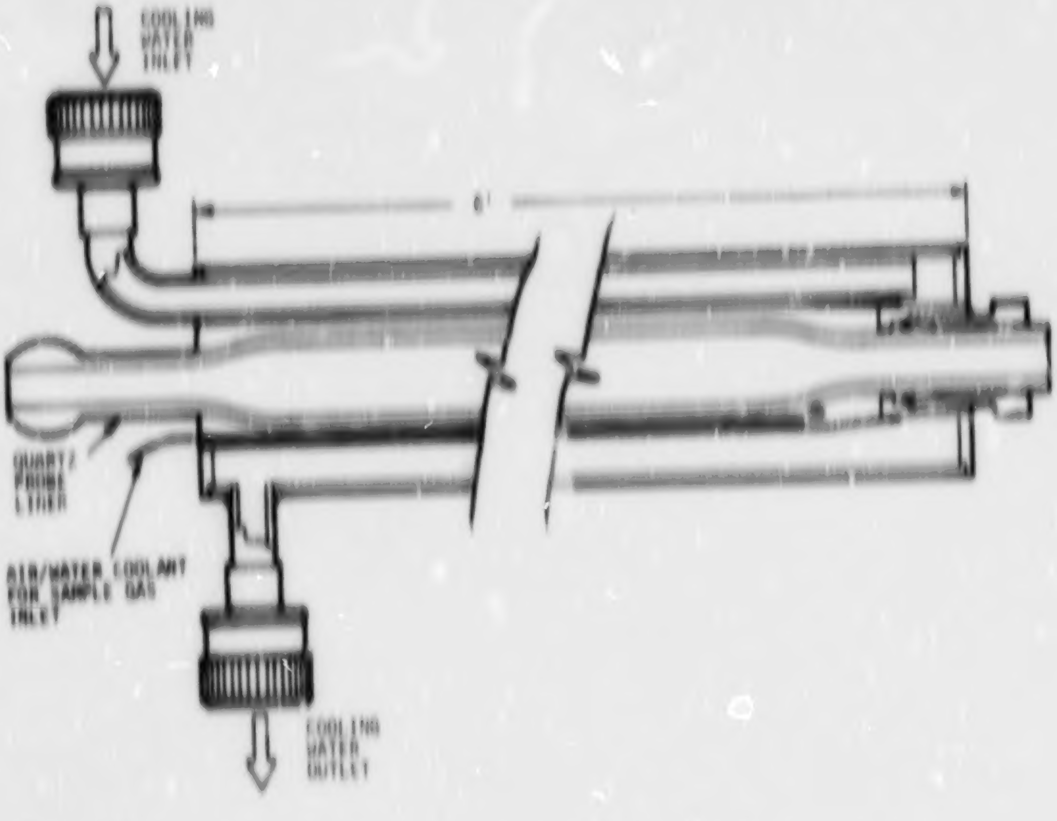

COOLING WATER INLET
6'
QUARTZ PROBE LINER
AIR/WATER COOLANT FOR SAMPLE GAS INLET
COOLING WATER OUTLET

Figure 4-6. Water-cooled probe design.

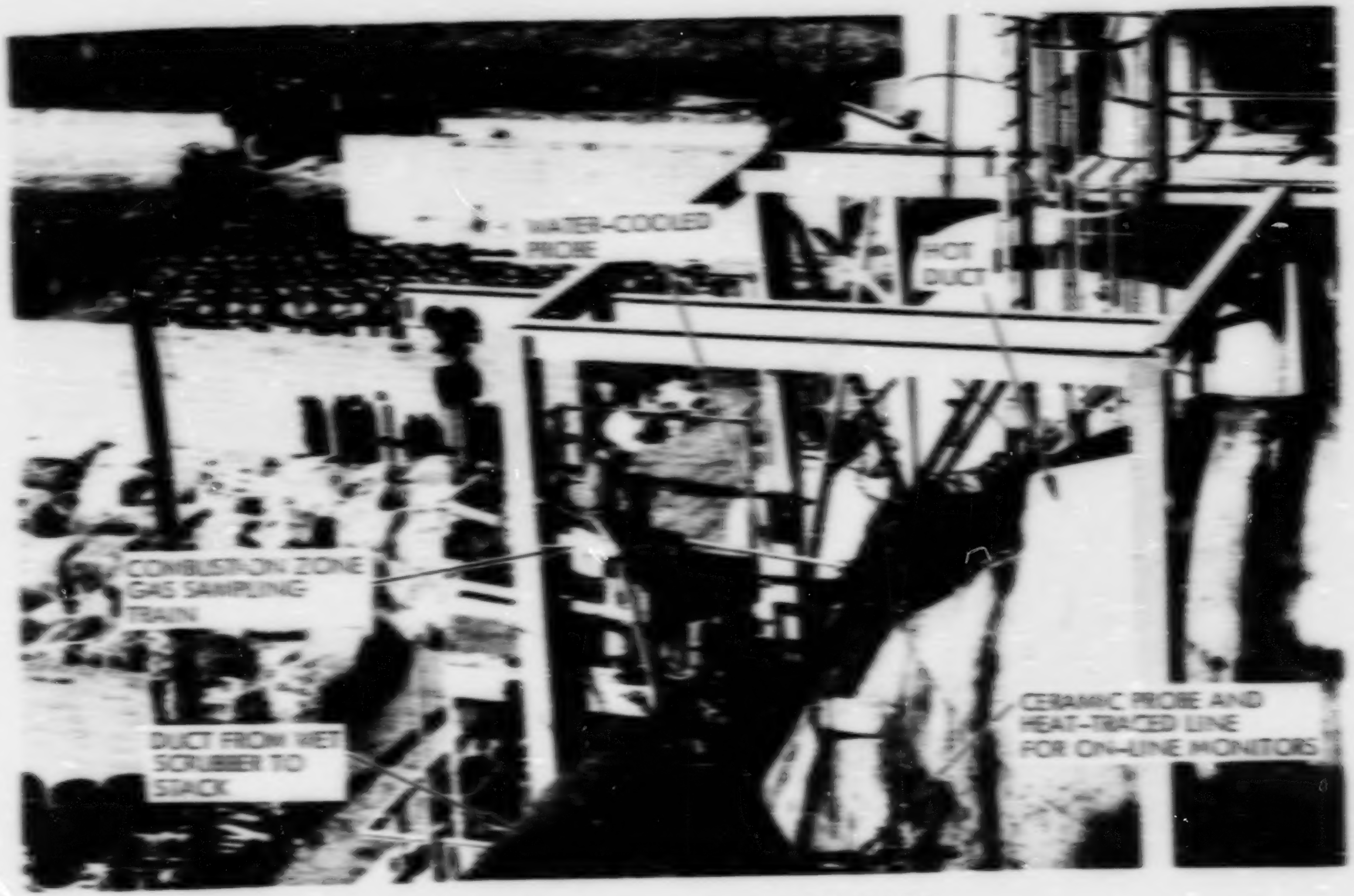

WATER-COOLED PROBE
HOT DUCT
COMBUSTION ZONE GAS SAMPLING TRAIN
DUCT FROM WET SCRUBBER TO STACK
CERAMIC PROBE AND HEAT-TRACED LINE FOR ON-LINE MONITORS

Figure 4-7. Combustion zone sampling site.

#### 4.1.3.3 Sampling Emissions and Waste Products

Samples of the stack effluent, spent scrubber water, and solid combustor residue were taken during and after each test to evaluate the environmental safety of the final emissions. An EPA Method 5 test was performed at the stack for particulate mass loading and composition determinations. Location of the sampling train at the test site is shown in Figure 4-8. Two diameters of the 2-meter diameter stack were traversed. Sampling was for one hour at approximately 20 liters/min. Gas volumes were measured to 0.03 liter, with a leak rate of less than 0.6 liter/min. Operating parameters for the train and sample volume data are tabulated in Appendix B.

Oxidizing agents ($H_2O_2$ and $(NH_4)_2S_2O_8$) were added to the impingers to aid scrubbing of trace metals. The following samples were obtained for each test from the stack sampling train:

- Acetone probe wash.
- 10-cm diameter particulate filter.
- Impinger solutions.
- Acidified split of impinger solutions.
- Spent silica gel.

Spent scrubber water samples were taken from the end of a drain pipe that carried the used scrubber water to treatment ponds. The fresh water being fed into the single pass wet scrubber was sampled from a pipe transporting the well water into the scrubber system.

The solid residues remaining after combustion of the wastes in the rotary kiln were accumulated in a hopper below the kiln throughout each test. Samples were then taken from this collected residue at the end of each test. The residue (consisting of steel hoops from the fiber drums, large chunks of metal, fine ash, etc.) was nonhomogeneous in nature, thus a truly representative sample could not be obtained without the use of grinding and milling equipment.

### 4.1.4 Analysis Techniques

Samples taken as described in Section 4.1.3 were analyzed for both organic and inorganic constituents. When necessary, extractions were performed first to concentrate the sample in a suitable form for analysis. Techniques used for these extractions and analyses will be briefly summarized here. For more detailed discussions, see the Rollins Analytical Plan (Reference 1).

#### 4.1.4.1 Extractions and Sample Preparation

Both solvents and acids were used to extract organics and inorganics, respectively, from the appropriate samples. These procedures and the basic sample preparation steps are listed in this section by sample type.

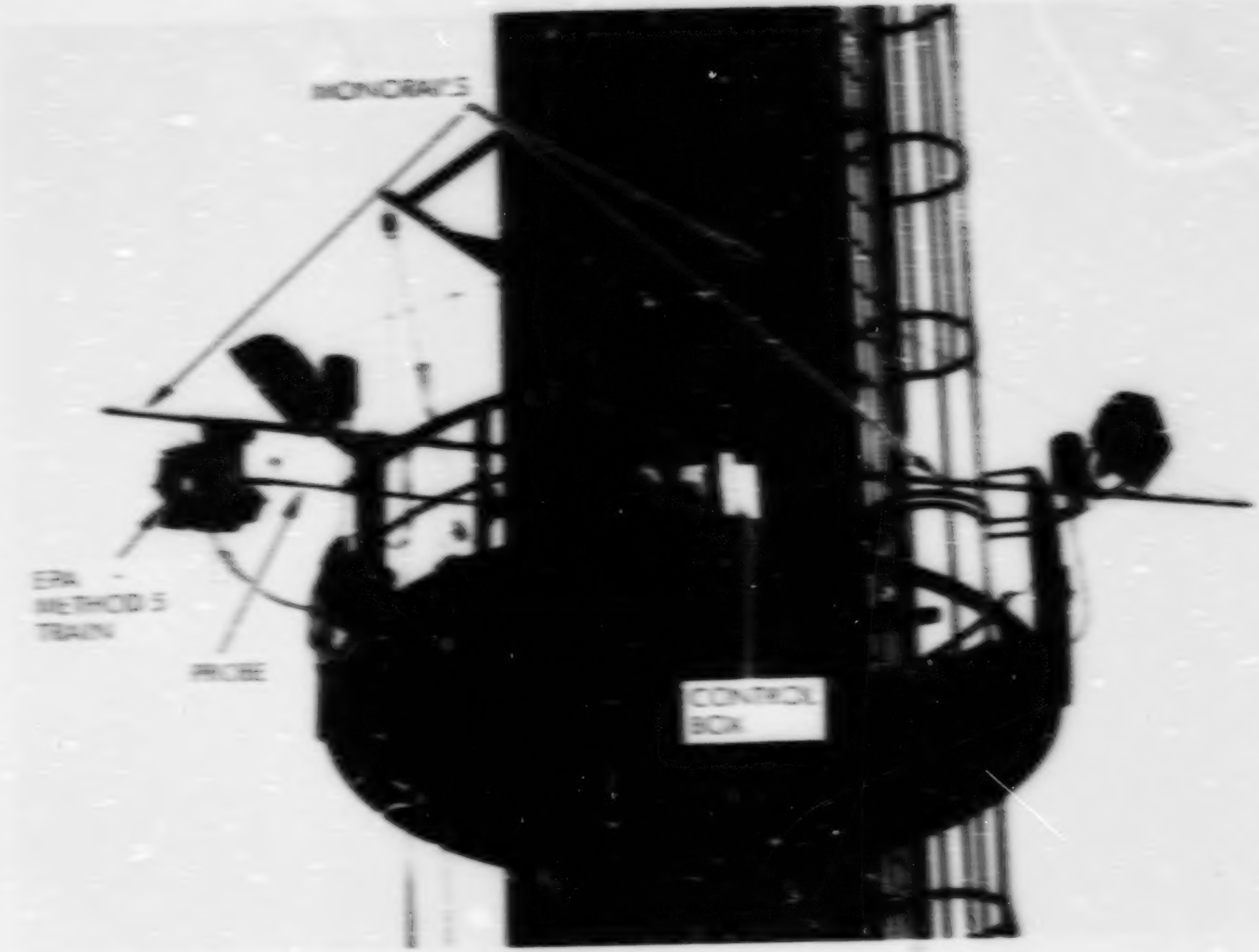

MONORAILS
EPA METHOD 5 TRAIN
PROBE
CONTROL BOX

Figure 4-8. Stack sampling site.

- Probe Washes

### Combustion Zone

The quartz liner had been rinsed first with acetone to remove organic matter. A water rinse was added to the procedure to remove the remaining particulate matter upon which the acetone had little effect. The acetone probe rinses were filtered. The solids were weighed and added to the filter from the sampling train in a Soxhlet apparatus. The acetone filtrate was added to the pentane extraction solvent. The combined filters were then extracted with this acetone/pentane mixture for 24 hours.

The particulate matter from the water probe rinses was recovered, dried and weighed.

### Stack

The acetone probe rinses were filtered through 5μ Teflon Millipore filters and the filtered solids weighed. This weight was added to the weight of the particulate matter on the filter for total mass loading calculations. The filtered solids were then combined with the particulate filters for acid extraction.

- Filters

### Combustion Zone

The tared sample filters plus one control were desiccated and weighed on consecutive days to a constant weight ±1 mg, combined with probe rinses, and then extracted in a Soxhlet apparatus for 24 hours with pentane. Solvent extracts were dried by being passed through a column of pre-extracted sodium sulfate and were then concentrated to 10 ml for analysis. At that time the filters were extracted with constant boiling aqua regia for six hours. This acid extract was made to 100 ml for analysis.

### Stack

The tared sample filters were weighed as for the combustion zone filters, combined with the solids filtered from the probe washes, and extracted with constant boiling aqua regia for six hours. The acid extracts were made to 100 ml for analysis.

- Solid Sorbent Traps

### Combustion Zone

The XAD-2 resin samples were transferred from the traps to glass Soxhlet thimbles and extracted in a Soxhlet apparatus with pentane and methanol for 24 hours with each solvent. These extracts were dried by being passed through a column of pre-extracted sodium sulfate and were then concentrated to 10 ml for analysis. One unused trap was extracted for background values and a blank on the solvent was also run. Another control was prepared by adding 0.1 mg of Aroclor 1242 to a trap and extracting as for the other samples.

Stack

No solid sorbent traps were used in the stack sampling train.

- Grab Gas

  Combustion Zone

  No special preparation was required.

  Stack

  No grab gas samples were taken at the stack.

- Impingers

  Combustion Zone and Stack

  The volume of liquid in the impingers was measured and the spent silica gel was weighed in the field after each test burn to determine the amount of water collected. The aqueous liquid impingers from the combustion zone were combined and 150 to 300 ml aliquots acidified in the field to stabilize the metals for analysis. The stack impinger samples were also acidified. No extractions or other special preparation steps were performed on any of the aqueous impinger samples.

  The acetone rinses of the combustion zone impinger glassware were combined and concentrated to a 10 ml volume for analysis.

- Scrubber Waters

  1.5 liter aliquots of the scrubber water samples were extracted for organics, according to the procedure for the separatory funnel extraction for oil and grease from water recommended by the EPA Handbook on Methods for Chemical Analyses of Water and Wastes, with the substitution of pentane for Freon (National Environmental Research Center, Cincinnati, Ohio 45268, EPA-625-/6-74-003). However, instead of evaporating the material to the dried residue, the extracts were concentrated to a 10 milliliter sample by use of a Kuderna-Danish concentrating evaporator. A control was also prepared by adding 0.2 mg of Aroclor 1242 to 1.5 liter of the fresh city water from Test 11 (PCB fluff). These scrubber water extracts were all dried before their concentration by being passed through a 200 x 10.5 mm glass column containing a 50 mm bed of sodium sulfate which had been pre-extracted with pentane in a Soxhlet for 24 hours.

- Solid Combustion Residues

  Approximately 100 g portions of the solid residue samples from the rotary kiln were extracted in a Soxhlet apparatus for 24 hours with pentane. The solvent extracts were dried and then concentrated to 10 ml for analysis. A control sample was prepared by adding 0.1 mg of Aroclor 1242 to 100 g of the residue from Test II (PCB fluff) and extracting with pentane in a Soxhlet.

  To prepare the residue samples for inorganic characterization, 100 mg aliquots were dissolved by a combination of digestion in a Teflon Parr bomb with aqua regia/HF, addition of boric acid, filtration, and ash and fusion of the remaining solids with sodium carbonate. The final sample volume was 100 ml. A reagent blank was also prepared.

### 4.1.4.2 Analytical Methods

After extraction of the samples for organic material and other preparation for inorganic material, the concentrated extracts, and aqueous solutions were analyzed by several methods which are summarized in Table 4-4. A general treatment of the sample preparation and analytical procedures is discussed below.

Organic Analyses. The concentrated solvent extracts of the filters, sorbent traps, scrubber waters, and solid residue samples were analyzed by gravimetry, IR, LRMS, and GC/MS techniques. An aliquot of each extract was evaporated at ambient conditions to remove the solvent. The residue was weighed and analyzed by IR and LRMS.

The IR and LRMS analyses yield qualitative information about the classes or types of compounds (e.g., hydrocarbons, phenols, POMs, PCBs, etc.) present as well as an idea of the complexity of the concentrated sample. Knowledge of the classes of compounds present provides a measure of the toxicity, if any, of the residue. The detection limits for these analytical techniques vary somewhat with the type of compound (see Table 4-4).

The grab gas samples contained in the Tedlar® film bags were analyzed by a mass spectrometer. A portion of the gas sample was vacuum transferred into the inlet system of constant volume and measurable pressure.

Quantitation of organic compounds, specifically PCBs, was performed by combined gas chromatography/mass spectrometry (GC/MS) using a Finnigan system. Detailed procedures for the analysis of PCBs are included in Appendix D.

Inorganic Analyses. Inorganic analyses were performed using inductively coupled plasma optical emission spectrophotometry (ICPOES). Samples from the acid extractions of the stack particulate filters, the acidified splits of the impingers and scrubber waters, and the dissolved solid residues were surveyed for trace metals by ICPOES. The ICPOES analysis determines

TABLE 4-4. SUMMARY OF ANALYTICAL METHODS

| Method | Instrument Manufacturer and Model | Detectability for a Compound or Element Being Sought |
|---|---|---|
| Organic Analyses | | |
| Gravimetry | Mettler, microbalance | 1 µg |
| Infrared Spectrophotometry (IR) | Perkin Elmer, 521 | ~3-5% of the sample being examined |
| Low Resolution Mass Spectrometry (LRMS) | Hitachi-Perkin Elmer, RMU-6 Mass Spectrometer | ~10 µg<br>(1% of a 1 mg sample) |
| Combined Gas Chromatography/Mass Spectrometry (GC/MS) | Finnigan, 9500 GC and Finnigan, 3300 Quadrapole Mass Spectrometer | ~100 ng per µl of sample |
| Inorganic Analyses | | |
| Inductively Coupled Plasma Optical Emission Spectrophotometry | Applied Research Laboratories, QA-137 | ~0.5-2000 ppb |
| Spark Source Mass Spectrophotography (SSMS) | AEI Scientific Apparatus Ltd., MS 702R | ~50-100 ppb |

32 elements, including most of the toxic elements of interest to the program, down to ppb levels with an accuracy of 10 to 100 percent.

Spark source mass spectrophotography (SSMS) was also used to analyze the solid residue samples for a broad survey of trace elements. This method is also sensitive to ppb levels and has an accuracy range of from 100 to 500 percent.

### 4.1.5 Problems Encountered

In spite of detailed planning and preparation for these tests, a few incidents occurred in the field that had not been anticipated. Minor problems were corrected immediately so the test schedule was not delayed. However, three incidents took place which either prevented or postponed testing; these are described in the following paragraphs. No problems were encountered in the analytical portion of the tests.

#### 4.1.5.1 Combustion Zone Sampling

An existing port in the hot duct was utilized as the sampling port for the combustion zone, by welding an 8-cm nipple to the port to accommodate the sampling probe. The immediate problem encountered was that the platform to be used by the sampling personnel was approximately two meters below the sampling port. Because of the windy weather conditions and the askew position of the welded nipple, it was impossible to safely support and operate the sampling probe and train. This problem was corrected by installing a unistrut monorail above the port and by adding an additional section of platform so that the sampling port was then only one meter above the platform. These corrective items caused a one-day delay in the testing.

#### 4.1.5.2 Storm Conditions

A storm front was moving in as the purge for the background test on No. 2 oil was begun. Conditions continued to worsen with high winds and heavy rain. Members of the sampling crew were just starting up the stack sampling train when lightning began striking tanks at nearby chemical plants. For the safety of the sampling personnel, it was then decided to forego acquiring a stack sample during the background test.

#### 4.1.5.3 Line Plugging

During the purge before the scheduled hammermilled fluff test, the line feeding the No. 2 oil to the kiln became plugged. The problem was corrected but not until it was too late in the day to run the test burn, so the test was postponed until the following day.

## 4.2 ADL TESTS OF NITROCHLOROBENZENE WASTE

### 4.2.1 Waste Tested

The waste which ADL tested at Rollins Environmental Services, Inc., (Rollins) was a stream generated in the production of nitrochlorobenzene (NCB). The principal use of the desired NCB product is as an intermediate material for the manufacture of synthetic dyes, drugs, pesticides, and photochemicals. The estimated total annual production of NCB waste similar to that selected for testing was 9100 metric tons (20 million lbs) in 1974.*

A survey sample of the NCB waste was received and analyzed by TRW, Inc., several months prior to testing. The survey analysis indicated that the waste was a black liquid with some suspended crystalline material, a specific gravity of 1.395 g/ml at 15.6°C, a higher heating value of 5050 Kcal/kg (9100 Btu/lb), and a low ash content (<0.001%). The elemental analysis of the sample was:

| | Weight Percent |
|---|---|
| Carbon | 47.6 |
| Nitrogen | 8.7 |
| Hydrogen | 3.0 |
| Chlorine | 20.6 |
| Sulfur | trace |

The major component of the waste was nitrochlorobenzene (mixture of isomers, 95% of total waste) with nitrobenzene as the next most abundant species. Inorganic elements identified at ppm levels were calcium, titanium, potassium, iron, antimony, silicon and phosphorus.

A second survey sample was obtained just prior to testing because the waste supplier indicated that the production process had changed. That sample was physically very different from the first. It contained much more crystalline material which had agglomerated into a mass of pale brown crystals which occupied about 50% of the volume of the sample container. The remainder of the sample was a black liquid. The mass of solid material had clearly formed in the sample container since it was several times larger than the mouth of the bottle. There was not sufficient time to do an extensive survey analysis on this second survey sample.

The waste actually tested was received at Rollins in a heated tank truck and was expected to resemble the second survey sample. However, the samples of representative waste feed drawn from the batch actually tested did not contain any noticeable quantity of crystalline material even after cooling. It is possible that the production process had been changed again, or that the second survey sample was not representative of the new waste, or that the waste within the tank truck was stratified.** (The crystalline material had been identified as p-chloronitrobenzene in the first survey analysis, and it is more dense than the ortho-isomer.) Whatever the reason, the waste material which was actually tested was found to be liquid at room temperature. Final analysis of the representative waste sample showed that it quite closely resembled the first survey sample.

---

* Destructing Chemical Wastes in Commercial Scale Incinerators, Technical Summary, Volume I, PB-257 709/6WP

** The entire contents of the tank truck were not required for testing because it was decided to blend the waste with diesel oil for destruction (see Sec. 4.2.5). The "representative" sample represents the material withdrawn from the truck for blending with the oil.

The details of the analyses of the representative waste sample were given in Appendix F, Section 4. The NCB waste itself was found to be about 95% by weight nitrochlorobenzene, primarily the ortho-isomer. Nitrobenzene and dinitrobenzene were also present, as well as some compounds tentatively identified as styrene-substituted di-tolylethers. The elemental analysis results, similar to those expected for nitrochlorobenzene, were:

| | Weight Percent |
|---|---|
| Carbon | 46.14 |
| Hydrogen | 2.80 |
| Nitrogen | 8.92 |
| Chlorine | 23.28 |
| Sulfur | 0.02 |

The ash content of the waste was estimated as 0.8%. No trace elements were found by SSMS at concentrations high enough to cause concern for emissions of toxic metals at the feed rates used in the tests.

Analysis of a sample of the diesel oil - NCB waste blend actually fed to the incinerator via the Loddby burner (see Section 4.2.5) confirmed that this mixture was 20% by volume or 28% by weight of nitrochlorobenzene. The blend was found to contain excess chlorine (10.0% Cl by weight total) compared to the levels calculated from its NCB content (6.92% by weight Cl calculated). No explanation for the apparent excess was determined, but the higher value was confirmed in three separate analyses and must be presumed to be real. Small amounts of PCBs (< 0.1%) were found in the blended waste - oil feed; these were found to be present in the #2 diesel oil used at Rollins and not in the NCB waste itself. The ultimate source of the PCBs was not traced (it may have been contamination of an oil truck, storage tank or the waste blend tank). That there is only a remote possibility of adverse environmental impact from incineration of these quantities of PCBs can be seen by consulting other sections of this report.

### 4.2.2 Operational Procedures

The preliminary detailed operating procedures including test plan and safety plan were reviewed prior to the arrival of the ADL sampling team on-site. Due to problems encountered in burning the NCB waste alone in the kiln, however, (these problems are described in more detail in Section 4.2.5.1) the NCB was dissolved in diesel oil and this solution was fired through the Loddby burner. For the test where the NCB waste alone was burned in the kiln (ADL Run R2), the kiln and afterburner were preheated and purged by burning diesel fuel (in the kiln and Loddby burner) for one hour prior to the feeding of the NCB waste. For the tests where NCB waste was mixed with diesel fuel and fed to the Loddby burner, there was no diesel fuel purge of the kiln and Loddby burner prior to firing the waste, since there was no means readily available for feeding straight diesel oil to the Loddby burner. (The only available feed tank for the Loddby burner was filled with the diesel fuel/NCB mixture.)

The operating procedure for the two test burns of the solution of NCB waste in diesel oil (ADL Test Runs R3 and R4) was as follows:

- The feed to the incineration system was switched over from commercial industrial waste being incinerated by Rollins to diesel fuel to the kiln and NCB waste/diesel oil blend to the Loddby burner (afterburner).
- The feed rates of oil and waste blend to the kiln and Loddby burner were adjusted to give the desired temperatures in the hot duct and afterburner.
- The on-line instruments were activated and test equipment and sampling trains were checked and prepared for use.
- After the NCB waste/diesel oil blend had been fed to the incinerator for at least one hour, the probes were inserted into the hot duct and stack and sampling begun.
- After the conclusion of sampling at the hot zone and stack for the day, the incineration system was switched over to other commercial wastes being incinerated by Rollins.

### 4.2.3 Sampling Methods

Sampling methods used in the NCB tests at Rollins are described briefly below.

Five distinct samples were taken during each waste test:

- Composite sample of waste feed material.
- Sample of combustion zone effluent fed to on-line instruments for continuous monitoring of test.
- Grab sample of combustion zone effluent to evaluate process effectiveness.
- Grab sample of stack gases to verify that test program was environmentally acceptable.
- Sample of fresh and spent scrubber water.

### 4.2.3.1 Waste Feed Sample

A composite sample of the blended waste-oil feed was obtained by collecting a portion of the material in the waste feed tank during each test. The two feed samples were blended to yield one representative sample.

### 4.2.3.2 On-line Gas Monitoring

A portion of the combustion effluent was sampled through a ceramic (12.5 mm) probe and passed through a heated Teflon line to a gas conditioning system. The gas conditioner was designed to deliver a cool, dry, particulate-free sample to the CO, $CO_2$, and $NO_x$ analyzers. A fraction of the sample was also supplied, untreated, to the hydrocarbon analyzer.

The instruments used and their ranges were:

| | | |
|---|---|---|
| Hydrocarbons | Beckman<br>Model 402 | 0.05 ppm - 10% |
| Carbon Monoxide | Beckman<br>Model P65 | 2-220 ppm |
| Carbon Dioxide | Beckman<br>Model 864 | 0.05 - 20% |
| Oxygen | Taylor<br>OA 273 | 0.05 - 100% |
| Nitrogen Oxides | Thermo Electron<br>Model 10A | 0.05 ppm -1% |

In addition, portions of this stream were sampled with gas-detector tubes.

### 4.2.3.3 Combustion Zone Sample

The train used for collecting this three-hour sample is shown schematically in Figure 4-9. The principal components in this comprehensive sampling train were:

- A 1.2 cm (0.5") quartz-lined sampling probe.
- A knockout trap consisting of an oversized impinger to condense water.
- A quartz fiber filter.
- A sorbent trap filled with XAD-2®* resin to collect organics of moderate volatility.
- Impingers containing aqueous sodium acetate to collect acidic gases. Sodium acetate, rather than sodium hydroxide, was used in the impingers to trap HCl. This was done to minimize the scrubbing of $CO_2$ that would tend to exhaust the capacity of the impinger solution and could also cause plugging problems by precipitation of sodium carbonate.

---

*Trademark of Rohm and Haas Company.

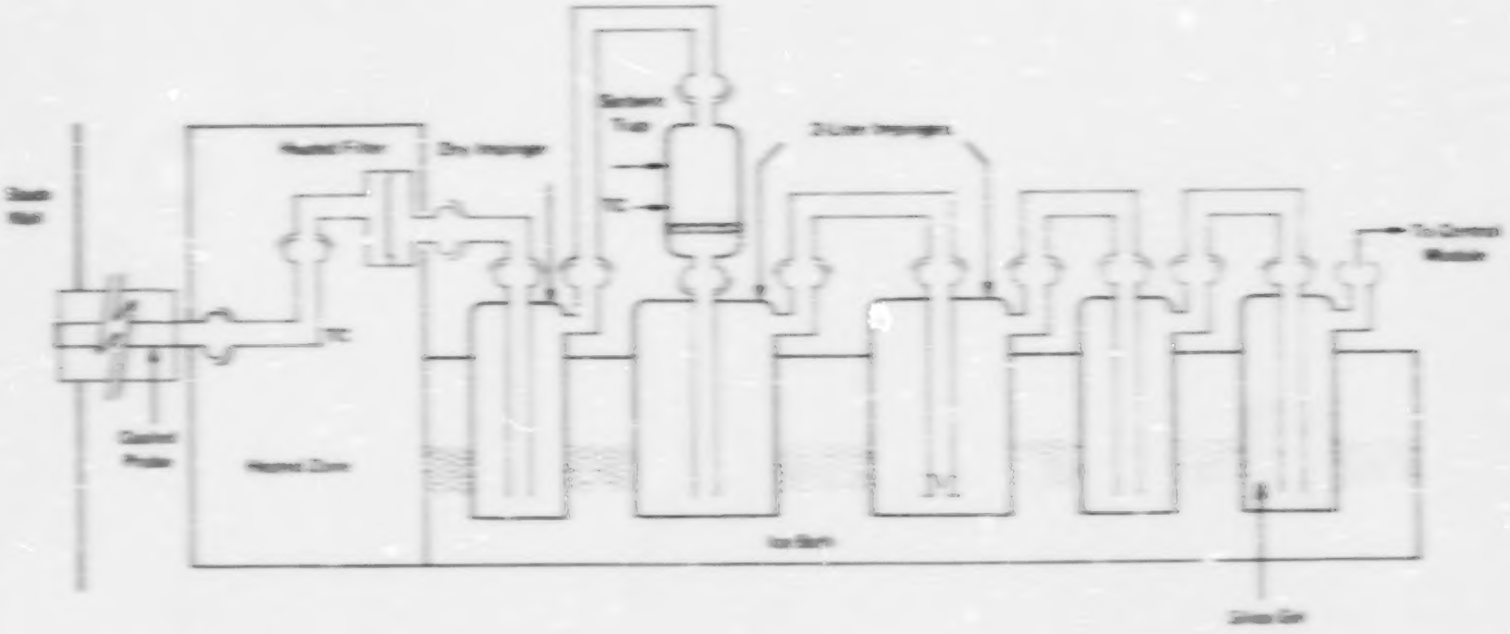

Stack Wall
Heated Filter
Dry Impinger
Sorbent Trap
TC
2-Liter Impingers
To Control Module
TC
Heated Zone
Ice Bath
Silica Gel

Figure 4-9. Hot zone sampling train for HCB tests.

In addition, a portion of the combustion zone effluent was collected in gas sampling bulbs from the bypass line of the hydrocarbon analyzer.

#### 4.2.3.4 Stack Gas Sample

The stack gas effluent was sampled isokinetically, according to the EPA Method 5 procedure, along two perpendicular traverses. The train was a typical EPA Method 5 type, the RAC Staksamplr.* The impingers contained aqueous sodium acetate to trap hydrochloric acid.

#### 4.2.3.5 Scrubber Water

Samples of spent scrubber water were compositud, using a proportional sampling pump, over a three-hour period coincident with the combustion zone gas grab sample. Samples of fresh scrubber water were taken in 1 liter bottles from a convenient tap.

### 4.2.4 Analysis Techniques

#### 4.2.4.1 Extractions and Sample Preparation

General descriptions of the techniques used are in the Phase I Final Report of this contract. A detailed description of the specific solvents, etc., used for the Rollins NCB test samples is given in Appendix F.

#### 4.2.4.2 Analytical Methods

The techniques which were chosen during Phase I of this contract for evaluation of the effectiveness of thermal destruction of industrial wastes were:

Low Resolution Mass Spectrometry (LRMS)
Infrared Spectrometry (IR)
Gas Chromatography
Elemental Analysis

Inorganic Analyses were done by:

X-ray Fluorescence (XRF)
Spark Source Mass Spectrometry (SSMS)
Atomic Absorption Spectroscopy (AAS)
Specific Ion Electrode Methods (SIE)

These techniques were applied to the Rollins NCB samples where appropriate. Details of analytical procedures are given in Appendix F.

---

*Trademark of Research Appliance Corporation.

### 4.2.5 Problems Encountered

#### 4.2.5.1 Process

In the first test burn of NCB waste (Test No. R2), the NCB waste alone was fed under pressure from a heated tank truck directly to the kiln and diesel fuel was fed to the Loddby burner to maintain temperature in the afterburner chamber. The NCB waste by itself would not atomize properly (in the air atomization nozzle in the kiln) and burned with considerable generation of carbon soot. The carbon soot was subsequently incinerated in the afterburner, but the NCB feed rate was limited to about 190 liters (50 gallons) per hour. Because of the poor feed rate and combustion of NCB in the kiln, it was decided to dissolve the NCB in diesel fuel and fire this solution through the Loddby burner which has more capacity than the kiln.

Approximately 20% by volume of NCB waste was blended with No. 2 diesel fuel in an agitated tank that feeds the Loddby burner. A laboratory test at Rollins indicated that up to 50% (by volume) of the NCB waste could be dissolved in the diesel oil. However, since the feed tank for the Loddby burner was about 80% full with diesel oil, enough NCB waste was added to fill the feed tank and give a 20% (vol.) solution of NCB in diesel oil.

#### 4.2.5.2 Sampling

The water pressure at the elevation of the hot zone probe was insufficient to supply adequate cooling water to the jacket of the hot zone probe. A booster pump was used to increase the water pressure, but the small diameter of the water lines to the jacket of the probe created so much back pressure at the water flow rate necessary to cool the probe (with 2-1/2 ft. of probe in the hot duct), that the pump motor would overheat and shut down. It was, therefore, necessary to back the probe out of the duct (so only about 1 foot remained in the duct) allowing the amount of cooling water to be reduced, thus preventing overheating of the pump.

#### 4.2.5.3 On-line Instruments

The oxygen analyzer failed when it was turned on the day before the R3 test. Trouble-shooting, after telephone consultation with the manufacturer's service representative, identified a defective analyzer cell as the most probable source of the symptoms observed. This could not be field repaired, and a replacement could not be obtained in time for completion of testing. For the R3 and R4 tests, the oxygen content in the combustion zone gas was estimated from the measured carbon dioxide concentration with the assumption that the sum of oxygen and carbon dioxide concentrations would be the same as for the background test.

The chemiluminescent NO/$NO_x$ analyzer appeared to be ineffective in the $NO_2$ to NO conversion mode. The instrument consistently gave the same concentration reading for the hot zone combustion gas whether in the NO mode or the $NO_x$ mode. Nitrogen dioxide was an expected product of combustion from the nitrochlorobenzene waste and, in fact, tests with gas detecting tubes which were on hand in the sampling trailer indicated more than 300 ppm of $NO_2$ in the combustion zone effluent. Telephone consultation with the manufacturer's service representative failed to produce an explanation for this phenomenon or to identify a remedy.

# 5. TEST RESULTS

As described in the previous section, the test burns at Rollins consisted of tests conducted by TRW on PCB-containing capacitors and tests conducted by ADL on nitrochlorobenzene manufacturing waste. The results obtained from these experiments are presented here in the following terms:

- Operating conditions and other data obtained in the field.
- Data from the analysis of the combustion gases for organic and inorganic species, particularly as relates to destruction of the waste. This will include a discussion of destruction efficiency for the ADL tests which had no solid residues left in the combustion zone.
- Data from analyses of the final emissions (i.e., stack gases, scrubber waters, and any solid residues from the kiln).

## 5.1 RESULTS OF PCB TESTS

### 5.1.1 Operating Conditions for Tests

The data presented in this section were collected from the operation of:

- Rollins rotary kiln incinerator facility, and
- TRW on-line gas composition monitors.

All recorded data for the incinerator system operating conditions were provided by Rollins and are summarized in Table 5-1. Temperatures in the rotary kiln system stayed fairly constant, with temperatures in the afterburner, for example, varying over a range of not more than ±40°C during any of the three tests.

Readings from all on-line gas monitors except the $SO_x$ analyzer were continuously recorded on strip charts. The resulting scans and readings were averaged over the 2-3 hour long test runs. Concentration values obtained for the dry combustion gas are shown in Table 5-2. HCl concentrations were determined by titration of the hot zone sampling train impingers.

Percent excess air was calculated according to the equation in EPA Method 3 (Reference 3) and was found to be 90, 84, and 91 percent for the respective tests. Percent moisture was calculated according to EPA Method 4 and was found to be 7.1, 6.5, and 8.5 percent in the combustion gas for the respective tests.

## TABLE 5-1. INCINERATOR SYSTEM PARAMETERS DATA SUMMARY

| | Test I | Test II | Test III |
|---|---|---|---|
| | No. 2 Oil Background | Hammermilled Capacitors | Whole Capacitors |
| **Flow Rates** | | | |
| No. 2 Oil to the Kiln (liters/min) | 34.8[a] | 4.48 | 9.34 |
| No. 2 Oil to the Loddby (liters/min) | | 35.7 | 29.0 |
| Natural Gas ($m^3$/min) | 0.989 | 1.51 | 1.46 |
| Waste (kg/hr) | - | 210[b] | 360 |
| Fresh Scrubber Water Feed (liters/min) | 3200 | 3200 | 3200 |
| Lime Slurry Feed (liters/min)[c] | 6.4 | 6.4 | 6.4 |
| **Temperatures** | | | |
| Kiln Flame (°C) | 1306 | 1252 | 1339 |
| Kiln Duct (°C) | 373 | 488 | 493 |
| Loddby Flame (°C) | 1485 | 1491 | 1509 |
| Afterburner (°C) | 1308 | 1331 | 1332 |
| Hot Duct (°C) | 1091 | 1089 | 1096 |
| Venturi Pressure Drop (cm $H_2O$) | 104 | 102 | 102 |
| Calculated Residence Time (sec) | - | 3.2 | 3.0 |

a) Only the total flow could be measured for this test. Subsequent changes separated the two feed systems, thus feed rates could also be measured separately.

b) This is the feed rate of the fluff material alone. The feed rate including the weight of the fiber drums was 330 kg/hr.

c) ∿30% solution by weight.

TABLE 5-2. GAS COMPOSITION DATA SUMMARY

| Test No. | $O_2$ (percent) | $CO_2$ (percent) | $N_2$[a] (percent) | CO (ppm) | $NO_x$ (ppm) | $SO_2$ (ppm) | HCl (ppm) | HC[b] (ppm) |
|---|---|---|---|---|---|---|---|---|
| I | 10.1 | 9.1 | 80.8 | 15 | 55 | 35 | - | <20 |
| II | 9.8 | 8.8 | 81.3 | 5-10 | 60 | 40 | 470 | <20 |
| III | 10.1 | 9.3 | 80.5 | 5-10 | 60 | 35 | 670 | <30 |

[a] By difference

[b] As methane

Attempts were also made to use Gastec® tubes to detect hydrocarbon species at the stack. However, because of the high moisture content of the stack gases and low ambient temperatures, the detection tubes became saturated with condensed water, so accurate readings could not be obtained. The condensation problem has been discussed in previous reports (Reference 2).

### 5.1.2 Composition of Combustion Zone Gas

Using the sampling train described in Section 4.1.3.2, samples of the combustion products were taken from the hot duct leading from the afterburner to the venturi scrubber. These samples were then separated into their organic and inorganic constituents and analyzed by appropriate techniques. Analysis of the combustion products was aimed mainly at identifying and quantifying any unburned waste material or hazardous partial combustion products. The production of potentially toxic levels of trace metals from burning these wastes was also examined. Where quantified species are calculated back to mg/m$^3$ in the sample gas stream, the gas volume data used is summarized in Appendix D.

#### 5.1.2.1 Organic Composition

Quantitation for Specific Compounds

The sample extracts were analyzed for specific toxic compounds found in the survey waste sample, i.e., PCB isomers, using a combined gas chromatography/mass spectrometer (GC/MS) method. The concentration level of interest for this program is defined as 0.1 mg/m$^3$ of sample gas, the threshold level of nearly all of the most hazardous species described in OSHA and other health and safety documents. However, currently there is much interest being given to PCB compounds, and it is known that they are considered hazardous at lower levels. Therefore, efforts were made to lower the detection levels for these specific compounds.

Two samples for each test were analyzed to determine PCBs in the combustion zone gas: 1) extracts of the particulate filter and probe wash and 2) extracts of the solid sorbent traps. No PCBs of any class were found in any of these samples. The minimum detectable quantities for the analysis were ~10 micrograms in the total 10 ml concentrated sample solution for each of the PCB classes. These minimum detectable quantities, when related to the average combustion zone sample gas volume of 1.9 $m^3$, represent about 5 micrograms per cubic meter (or 0.005 $mg/m^3$) for the filter and sorbent trap samples combined.

A recovery study was conducted by adding 0.100 mg of Aroclor 1242 to an unused XAD-2 sorbent trap as a doped control. The doped trap was then extracted and the extract treated along with the test sample extracts. Analysis of the doped control extract by GC/MS found 0.2 mg of total PCBs, indicating a recovery of 200 percent. This difference between added PCBs and recovered PCBs was within the expected accuracy range for PCBs at these low levels.

Qualitative Surveys

Combustion zone samples were also surveyed by gravimetric, infrared spectrometry (IR), and low resolution mass spectrometry (LRMS) techniques. The qualitative results are discussed in the following order:

- Combined particulate filter and probe wash extracts
- Sorbent trap extracts
- Acetone impinger rinses
- Grab gas samples

Combined Probe Wash and Particulate Filter Extracts. The types of organic material discovered in this survey are typical of those compounds found in most of the other organic residues obtained in the analyses from these tests. Hydrocarbon oil, dioctyl phthlate, and silicone were the predominant species present in these samples. In addition, traces of palmitic acid, stearic acid, substituted aromatics, and an unknown substance with a molecular weight of 382 were identified. Table 5-3 shows the amounts of the various materials that were present. The values have been corrected for those obtained from blanks and controls which were considerably lower.

Sorbent Traps. The amounts of material extracted from the sorbent traps and found as a residue after mild evaporation are presented in Table 5-4. The quantities have been corrected for the unused, control sorbent trap extract. The types of matter found in the trap extracts by IR and LRMS analyses were essentially the same for all samples. The major compound found in all three test samples was silicone, with minor amounts of hydrocarbon oil and substituted aromatics. In addition, traces of a low molecular weight amine and a glycol ether were present in the extract samples from Tests I and II, respectively.

## TABLE 5-3. SUMMARY OF SURVEY ANALYSIS ON THE COMBINED PROBE WASH AND PARTICULATE FILTER EXTRACTS

| Test No. | Amount of Material Found as Residue[a] (mg) | Volume of Sampled Gas[b] ($m^3$) | Concentration in Sample Gas ($mg/m^3$) |
|---|---|---|---|
| I | 15.4 | 3.65 | 4.21 |
| II | 38.2 | 3.88 | 9.84 |
| III | 76.0 | 3.88 | 19.6 |

[a] Corrected for blank on extraction glassware and solvent.

[b] Includes water vapor.

## TABLE 5-4. SUMMARY OF SURVEY ANALYSIS ON SORBENT TRAP EXTRACTS

| Test No. | Material Extracted by Pentane (mg)[a] | Volume of Sample Gas[b] ($m^3$) | Concentration of Extractables in Sample Gas ($mg/m^3$) |
|---|---|---|---|
| I | 34.1 | 3.65 | 9.3 |
| II | 16.9 | 3.88 | 4.4 |
| III | 12.8 | 3.88 | 3.3 |
| Doped Control[c] | 1.23 | - | - |

[a] Corrected for blank control sorbent trap extract weight.

[b] Includes water vapor.

[c] An unused trap with 0.100 mg of Aroclor 1242 added.

The primary purpose of preparing the doped control sample was to provide recovery data for the quantitative organic analysis. Thus, the residue obtained from this sample was not qualitatively analyzed beyond the gravimetric determination.

*Acetone Impinger Rinses.* As an added check for PCBs on the combustion zone sampling train, the impinger glassware was rinsed with acetone at the end of each test. For maximum sensitivity the rinses were combined, concentrated, and qualitatively examined for PCBs. The IR and LRMS analyses of this sample found only silicone and dioctyl phthlate. There was no indication of PCBs.

*Grab Gas Samples.* The contents of the Tedlar® gas sampling bags were analyzed by introducing a portion of the gas into the mass spectrometer and measuring its pressure at constant volume. Low molecular weight hydrocarbons were detected at ppm levels, but no PCBs were disclosed.

### 5.1.2.2 Inorganic Characterization

Inorganic elemental concentrations were determined by analysis of the particulate filters and aqueous impinger samples. Figure 5-1 shows a photograph of the particulate filters obtained from sampling the combustion gases from Tests I through III (from left to right in the top row). Trace metals on the particulate filters were put into solution by acid digestion of the filters. The filter digests and the impinger solutions were then surveyed by ICPOES to determine the major elements. The results of this analysis are shown in Table 5-5. The major elements found in these samples were boron, cadmium, copper, iron, potassium, phosphorus, lead, and tin. Major levels of aluminum may also have been present; however the high background of this element in the glass fiber filters makes that impossible to determine. Other components which were analyzed by the ICPOES study but were undetected in combustion zone samples are shown in Table 5-6.

### 5.1.3 Final Emissions

Emissions from the Rollins rotary kiln process were sampled and analyzed to evaluate the environmental safety of the waste burns. All of the final process effluents were sampled; these included stack gas, scrubber water, and solid residue.

### 5.1.3.1 Stack Gas

Stack effluents were sampled during the tests with a standard EPA Method 5 train. The samples obtained were analyzed to determine particulate loading in the effluent gas and elemental composition of the particulate matter. Figure 5-1 shows the particulate filters obtained from sampling the stack gas from Tests II and III (from left to right in the bottom row). No stack sample was procured from Test I for the reasons discussed in Section 4.1.5.

Particulate loading was determined by adding the weight gain on the filters to the weight of residue in the probe washes. This total was then divided by the dry sample gas volume. The loading values acquired are listed in Table 5-7.

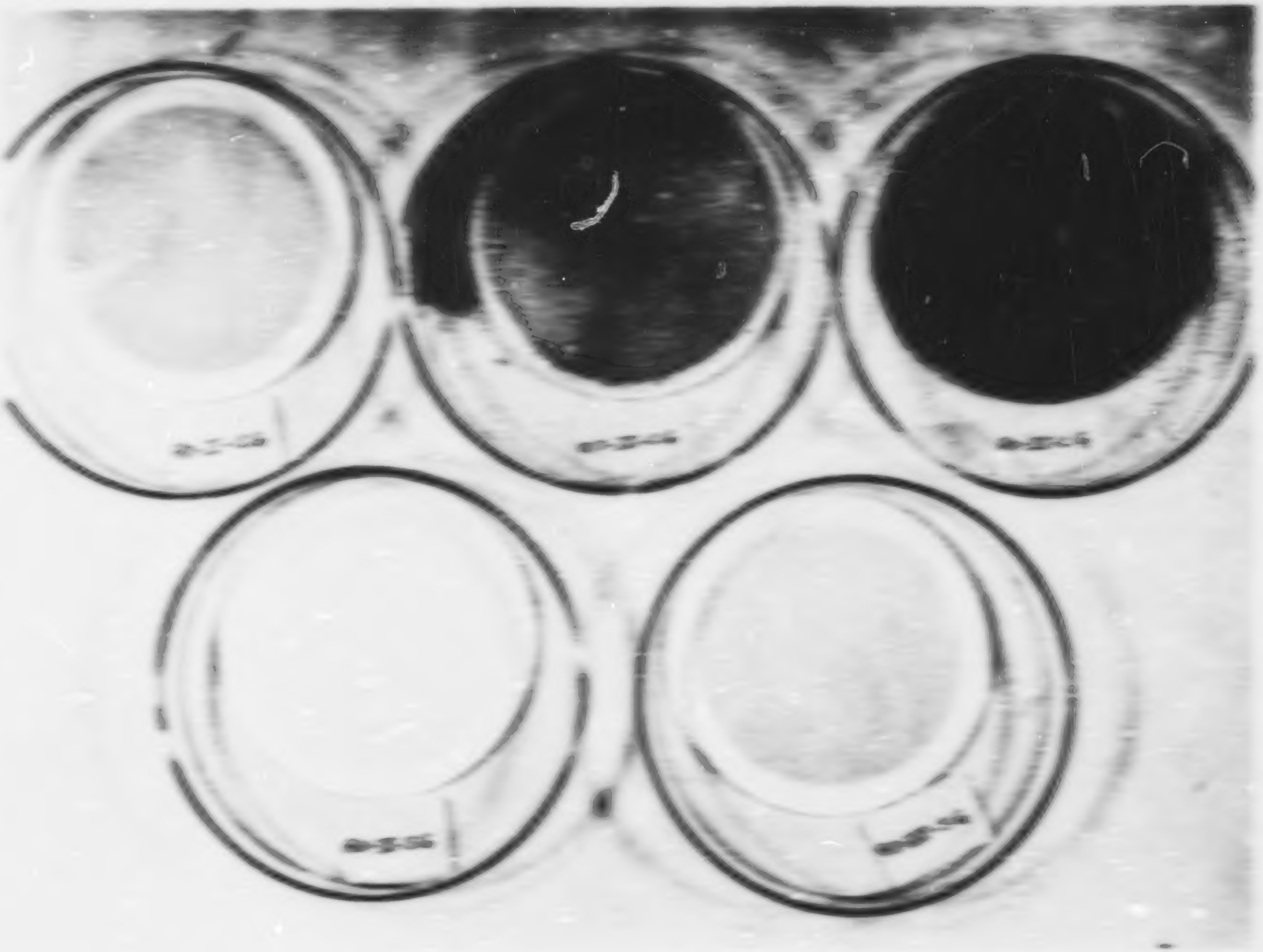


Figure 5-1. Filters from combustion zone and stack gas sampling trains.

## TABLE 5-5. SURVEY FOR TRACE METALS IN COMBUSTION ZONE SAMPLES BY ICPOES

| Element | Concentration in the Sample Gas (mg/m$^3$) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Test I | | | Test II | | | Test III | | |
| | Filter | Impingers | Total | Filter | Impingers | Total | Filter | Impingers | Total |
| Al | ≤0.94[a] | 0.074 | ≤1.0 | ≤51 | ND(0.002) | ≤50 | ≤7.3 | ND(0.004) | ≤7 |
| As | 0.001 | ≤0.003 | ≤0.004 | 0.006 | ND(0.00003) | 0.006 | 0.074 | ≤0.005 | 0.08 |
| B | ≤0.46 | 0.77 | ≤0.6 | ≤0.66 | 36 | ≤4 | ≤0.72 | 0.36 | ≤0.3 |
| Ba | ≤0.74 | ND(0.036) | ≤0.7 | ≤0.075 | ND(0.030) | ≤0.1 | ≤0.046 | ND(0.036) | ≤0.3 |
| Be | ND(0.00002) | ≤0.0002 | ≤0.0002 | ≤0.0003 | ND(0.00001) | ≤0.0003 | ND(0.00001) | ≤0.0004 | ≤0.0007 |
| Ca | ≤0.88 | ≤0.036 | ≤0.9 | 2.2 | ≤0.007 | 2 | ≤0.54 | ≤0.028 | ≤0.5 |
| Cd | 0.007 | ND(0.007) | ≤0.002 | 0.16 | ND(0.005) | 0.2 | 0.70 | ND(0.002) | 0.7 |
| Co | 0.003 | ND(0.002) | ≤0.003 | 0.002 | ND(0.002) | ≤0.004 | 0.002 | ND(0.002) | ≤0.004 |
| Cr | 0.01 | 0.005 | 0.02 | 0.025 | ND(0.0006) | 0.02 | 0.056 | 0.72 | 0.02 |
| Cu | 0.036 | ≤0.002 | 0.04 | 2.2 | ND(0.00006) | 2 | 8.3 | 0.007 | 8 |
| Fe | 1.3 | ≤0.040 | ≤1.4 | 72 | ≤0.072 | 72 | 72 | ≤0.005 | 80 |
| K | ND(1.6) | ND(0.22) | ND(1.8) | 7.1 | ND(0.78) | 7 | 0.85 | ND(0.22) | 1 |
| Mg | ≤0.26 | ND(0.00002) | ≤0.3 | ≤0.29 | ND(0.00001) | ≤0.3 | ≤0.16 | ND(0.00002) | ≤0.2 |
| Mn | ≤0.047 | 0.0009 | ≤0.054 | ≤0.054 | ND(0.00004) | ≤0.05 | ≤0.042 | ND(0.00008) | 0.04 |
| Mo | 0.011 | 0.005 | 0.008 | 0.015 | 0.07 | 0.2 | 0.07 | 0.056 | 0.07 |
| Na | ≤3.4 | —[b] | ≤4 | ≤0.7 | —[b] | ≤6 | ≤0.7 | —[b] | ≤4 |
| Ni | 0.033 | ND(0.007) | 0.07 | 0.033 | ND(0.0004) | 0.03 | 0.024 | ND(0.007) | 0.02 |
| P | 0.092 | ND(0.058) | ≤0.7 | 7.90 | ND(0.746) | 7 | 1.7 | 0.77 | 2 |
| Pb | 0.36 | ND(0.009) | 0.4 | 72 | ND(0.028) | 70 | 31 | ND(0.010) | 30 |
| Si | ≤0.38 | 2.6 | 3 | ≤0.34 | 0.67 | ≤0.9 | ≤0.39 | 0.60 | 1 |
| Sn | ND(0.002) | ND(0.005) | ND(0.007) | 0.88 | 0.083 | 1 | 2.6 | ND(0.005) | 3 |
| Sr | ≤0.028 | ND(0.0007) | ≤0.009 | ≤0.006 | ND(0.00006) | ≤0.006 | ≤0.005 | ND(0.0005) | ≤0.005 |
| Ti | 0.016 | 0.006 | 0.02 | 0.33 | ≤0.005 | 0.3 | 0.017 | ≤0.002 | 0.02 |
| V | 0.003 | 0.0003 | 0.003 | 0.77 | ND(0.00006) | 0.7 | 0.071 | ND(0.00008) | 0.07 |
| Zn | ≤0.72 | 0.002 | ≤0.7 | ≤2.3 | 0.083 | ≤2 | ≤0.37 | 0.02 | ≤0.4 |

[a] ≤, a less than or equal to sign indicates those elements which were detected but not significantly above background levels.

[b] Not appropriate because NaOH was added to these impingers.

TABLE 5-6. DETECTION LIMITS FOR ELEMENTS NOT FOUND IN THE COMBUSTION ZONE SAMPLES BY ICPOES

| Element | Average Detection Limit (mg/m$^3$) | | |
|---|---|---|---|
| | Filter | Impingers | Total |
| Au | 0.0002 | 0.0005 | 0.0007 |
| As | 0.001 | 0.004 | 0.005 |
| Eu | 0.0004 | 0.002 | 0.002 |
| Se | 0.002 | 0.006 | 0.008 |
| Te | 0.002 | 0.007 | 0.009 |
| U | 0.002 | 0.008 | 0.01 |
| W | 0.003 | 0.009 | 0.01 |

TABLE 5-7. PARTICULATE LOADING IN THE EFFLUENT GAS

| Test | Weight on Filter (mg) | Weight in Probe Wash (mg) | Total Weight (mg) | Sample Gas Volume, Dry (m$^3$) | Particulate Loading | |
|---|---|---|---|---|---|---|
| | | | | | mg/m$^3$ | Grains/scf |
| II-"Fluff" | 44.5 | 9.0 | 53.5 | 1.55 | 35 | 0.015 |
| III-Whole Capacitors | 33.6 | 21.5 | 55.1 | 1.04 | 53 | 0.022 |

After weighing, the filters were acid digested and along with the impinger solutions were surveyed for trace metals by inductively coupled argon plasma optical emission spectroscopy (ICPOES). The results of this survey are shown in Table 5-8. The predominant elements found in the combustion gas were also the major inorganic constituents of the stack gas. Copper, iron, lead, phosphorus, tin, and cadmium were the major elements in the stack, out of which only lead and cadmium are considered to be significantly toxic. Of the 32 elements that were determined by the

## TABLE 5-8. SURVEY FOR TRACE METALS IN STACK SAMPLES BY ICPES

| Element | Concentration in the Stack Gas (mg/m³) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Test II | | | | Test III | | | |
| | Filter | 1st Impinger | 2nd Impinger | Total | Filter | 1st Impinger | 2nd Impinger | Total |
| Al | ≤0.14[a] | 0.078 | 0.009 | ≤0.2 | ≤1.44 | ND (0.013) | 0.031 | ≤1.5 |
| As | 0.003 | 0.063 | [b] | 0.066 | 0.070 | 0.013 | [b] | 0.08 |
| B | ≤0.087 | 0.55 | 0.17 | ≤0.8 | ≤0.30 | 0.52 | 0.040 | ≤0.9 |
| Ba | ≤0.021 | ND (0.0011) | 0.15 | ≤0.2 | ≤0.26 | ND (0.0020) | ND (0.006) | ≤0.3 |
| Be | ≤0.00003 | 0.00008 | ND (0.00002) | ≤0.0001 | ≤0.00004 | 0.0002 | ND (0.00012) | 0.0002 |
| Ca | ≤0.64 | ND (0.0071) | 0.023 | ≤0.7 | ≤0.88 | ND (0.002) | 0.055 | ≤0.9 |
| Cd | 0.14 | ND (0.003) | ND (0.002) | 0.1 | 0.40 | ND (0.004) | 0.007 | 0.4 |
| Co | ND (0.011) | ND (0.004) | 0.021 | ≤0.03 | ND (0.002) | ND (0.006) | 0.020 | ≤0.02 |
| Cr | 0.032 | ND (0.002) | ND (0.001) | 0.03 | 0.038 | ND (0.002) | ND (0.001) | 0.04 |
| Cu | 0.27 | ND (0.0007) | 0.002 | 0.3 | 7.5 | 0.014 | 0.012 | 8 |
| Fe | 1.6 | ≤0.034 | ≤0.014 | 2 | 5.5 | ≤0.032 | ≤0.036 | 6 |
| Hg | ≤0.26 | ND (0.00002) | ND (0.00005) | ≤0.3 | ≤0.29 | ND (0.0001) | ND (0.00005) | ≤0.3 |
| Mg | ≤0.096 | ND (0.0003) | ND (0.0002) | ≤0.096 | ≤0.02 | ND (0.0004) | ND (0.0002) | ≤0.02 |
| Mn | 0.074 | 0.036 | ND (0.007) | 0.1 | 0.16 | 0.077 | ND (0.002) | 0.2 |
| Na | ≤5.4 | ND (0.16) | 0.52 | ≤7 | ≤6.3 | 0.79 | 0.90 | ≤8 |
| Ni | 0.039 | ND (0.003) | ND (0.002) | 0.02 | 0.059 | ND (0.004) | ND (0.002) | 0.06 |
| P | 0.17 | ND (0.15) | ND (0.07) | ≤0.4 | 1.9 | ND (0.2) | ND (0.07) | 2 |
| Pb | 2.7 | ND (0.004) | ND (0.016) | 3 | 3.3 | ND (0.005) | ND (0.014) | 3 |
| Sb | ≤0.40 | 0.22 | 0.70 | ≤1 | ≤1.7 | 0.17 | 0.60 | ≤3 |
| Se | 0.72 | ND (0.013) | ND (0.004) | 0.7 | 0.72 | ND (0.002) | ND (0.002) | 0.7 |
| Sr | ≤0.003 | ND (0.0007) | ND (0.0002) | ≤0.003 | ≤0.011 | ND (0.0004) | ND (0.0004) | ≤0.01 |
| Ti | 0.087 | 0.002 | 0.002 | 0.09 | 0.17 | 0.002 | 0.003 | 0.2 |
| V | 0.056 | ≤0.008 | ND (0.0007) | ≤0.07 | 0.077 | ≤0.0072 | ND (0.0004) | ≤0.08 |
| Zn | ≤2.1 | 0.015 | ND (0.0004) | ≤2 | ≤0.96 | 0.028 | 0.003 | ≤1 |

[a] ≤, a less than or equal to sign indicates those elements which were detected but not significantly above background levels.

[b] Not appropriate because $HNO_3$ was added to this impinger.

ICPOES analysis, eight were not detected in the stack samples. These eight components are listed in Table 5-9, with a calculation of the average detectable limit for each in the flue gas.

### 5.1.3.2 Scrubber Water

The Rollins scrubbing system consisted of an essentially single pass, wet scrubber. Samples were obtained for each of the three tests of both the well water feed (FSW) and the spent water (SSW) exiting the system. Aliquots of these samples were both solvent extracted for analysis of organics and acidified to stabilize inorganics for analysis. Comparison of the analytical results for the FSW and SSW samples yields an estimate of how much, if any, hazardous species were added to the scrubber water during each test.

#### Organic Composition

As with other samples for analysis of organics, the scrubber water extracts were quantitatively analyzed for specific compounds and qualitatively surveyed for overall composition.

Quantitative Results. The solvent extracts of the scrubber water samples were concentrated and examined for PCBs by GC/MS. One city water input and all three effluent water samples were analyzed. No PCBs of any class were found in any of the samples at a minimum detection limit of 0.007 mg/liter.

TABLE 5-9. DETECTION LIMITS FOR ELEMENTS NOT FOUND IN THE STACK SAMPLES BY ICPOES

| | Average Detection Limit (mg/m$^3$) | | | |
|---|---|---|---|---|
| | | Impingers | | |
| Element | Filter | First | Second | Total |
| Au | 0.0004 | 0.002 | 0.0008 | 0.003 |
| As | 0.003 | 0.01 | 0.007 | 0.02 |
| Eu | 0.001 | 0.005 | 0.002 | 0.008 |
| K | 0.2 | 0.7 | 0.3 | 1 |
| Se | 0.006 | 0.02 | 0.01 | 0.04 |
| Te | 0.005 | 0.02 | 0.01 | 0.04 |
| U | 0.006 | 0.03 | 0.01 | 0.05 |
| W | 0.007 | 0.03 | 0.01 | 0.05 |

A recovery study was also conducted wherein 0.200 mg of Aroclor 1242 was added to 1.5 liters of the input well water sample from Test II. The doped sample was then extracted and the extract treated along with the test sample extracts. GC/MS analysis of the doped sample found 0.13 mg of total PCBs, yielding a recovery of 65 percent. This indicates that the combined effect of recovery efficiency and analytical limits was adequate to detect PCBs down to a level of 0.01 mg/liter.

*Qualitative Survey Data.* Aliquots of the solvent extracts were evaporated at ambient conditions. The residue obtained from this step was then weighed and analyzed by IR and LRMS techniques. The concentration of extractable species in the scrubber waters, as calculated from the extract residue weights, is presented in Table 5-10. The data from the IR analysis indicate that silicone was the major component of the residues. The residues from these samples were generally quite low.

The doped control sample was prepared primarily for recovery data for quantitative analyses. For this reason the residue from this sample was not qualitatively analyzed beyond obtaining the gravimetric value.

## Inorganic Characterization

The acidified aliquots of the scrubber water samples were surveyed by ICPOES to determine the major elements present. The results of this analysis are shown in Table 5-11. Substantial increases were observed for aluminum, copper, lead, and zinc compared to the concentrations in both the input city water and the effluent from the background test (Test I). Elements not detected by the ICPOES investigation in any of the scrubber water samples are listed in Table 5-12 along with their detection limits.

TABLE 5-10. SUMMARY OF SURVEY ANALYSIS OF SCRUBBER WATER EXTRACTS

| Test and Sample Identification | Volume of Water Extracted (liters) | Weight of Residue in Extract (mg) | Concentration in Scrubber Sample (mg/liter) |
|---|---|---|---|
| I - FSW | 1.5 | 0.24 | 0.16 |
| SSW | 1.5 | 0.29 | 0.19 |
| II - SSW | 1.5 | 0.28 | 0.19 |
| III - SSW | 1.5 | 0.35 | 0.23 |
| Doped Control[a] | 1.5 | 1.07 | - |

[a] Water with a known amount of Aroclor 1242 added.

TABLE 5-11. SURVEY FOR TRACE METALS IN SCRUBBER WATER SAMPLES BY ICPOES

| Element | Concentration in Scrubber Water (mg/liter) | | | | |
|---|---|---|---|---|---|
| | FSW | | SSW | | |
| | Test II | Test III | Test I | Test II | Test III |
| Al | ND(<0.04) | 0.09 | 1.7 | 3.0 | 4.7 |
| B | 0.15 | 0.16 | 0.20 | 1.6 | 0.24 |
| Ba | ND(<0.05) | ND(<0.05) | 0.23 | 0.14 | ND(<0.05) |
| Be | ND(<0.001) | ND(<0.001) | 0.013 | 0.003 | 0.003 |
| Ca | 23 | 24 | 860 | [illegible] | 600 |
| Cu | 0.007 | 0.010 | 0.089 | 0.15 | 7.3 |
| Fe | 0.065 | 0.10 | 2.4 | 0.50 | 1.7 |
| K | 30 | 30 | 120 | 60 | 100 |
| Mg | 5.0 | 5.1 | 12 | 6.4 | 8.5 |
| Mn | 0.055 | 0.055 | 0.16 | 0.048 | 0.10 |
| Na | 99 | 100 | 120 | 110 | 120 |
| Ni | 0.05 | 0.05 | 0.12 | 0.04 | 0.05 |
| P | 1.1 | ND(<0.5) | 2.1 | 1.4 | 3.0 |
| Pb | ND(<0.09) | ND(<0.09) | ND(<0.09) | 1.7 | 4.0 |
| Si | 11 | 12 | 17 | 12 | 15 |
| Sr | 0.20 | 0.20 | 0.72 | 0.40 | 0.58 |
| Ti | 0.010 | 0.009 | 0.063 | 0.020 | 0.031 |
| V | 0.007 | 0.005 | 0.042 | 0.028 | 0.025 |
| Zn | 0.05 | 0.03 | 0.16 | 1.5 | 1.3 |

TABLE 5-12. DETECTION LIMITS FOR ELEMENTS NOT FOUND IN THE SCRUBBER WATERS BY ICPOES

| Element | Detection Limit (ppb) | Element | Detection Limit (ppb) |
|---|---|---|---|
| Ag | 0.3 | Mo | 11 |
| As | 40 | Se | 60 |
| Au | 8 | Sn | 50 |
| Cd | 11 | Te | 65 |
| Co | 16 | U | 80 |
| Cr | 6 | W | 90 |
| Eu | 15 | | |

### 5.1.3.3 Solid Residue

The objective of analyzing the solid residue samples taken at the end of the tests was to determine whether any residual hazardous materials were present which would affect disposal methods or which would in themselves constitute an environmental hazard. Figure 5-2 presents a photograph of the two solid residue samples. They were quite similar in physical appearance except that the Test II sample was slightly reddish in color.

Organic Composition

Portions of the residue samples were extracted in a Soxhlet apparatus with pentane. The resulting extracts were then analyzed by the quantitative and qualitative methods described previously.

Quantitative Data. The results of the GC/MS analysis for PCB compounds in the solid residue extracts are presented in Table 5-13. PCBs were found in the Test III residue (whole capacitor feed) at a level of 470 mg/kg total PCBs, but were undetected in the Test II residue (hammermilled fluff feed). The lower limit of detection for PCBs for this study corresponded to 0.1 mg/kg or 0.1 ppm by weight. The distribution of PCBs in the Test III sample over the five classes found is significantly different from that of the waste feed material (see Table 4-2). The differing profiles indicate a much higher destruction efficiency for the lower chlorinated PCBs and a decreasing efficiency as the number of chlorine atoms per PCB molecule increases.

A doped control was also prepared by adding 0.100 mg of Aroclor 1242 to ~0.1 kg of the residue from Test II. This doped sample was then extracted and the extract treated in the same manner as the test samples. Analysis of the doped sample extract did not detect the presence of any PCBs, indicating a recovery efficiency of less than ten percent. Thus the extracted PCB quantity for the Test III sample could actually be as much as 4,700 ppm (~0.5 percent) or higher. The detectable limit in the Test II sample is probably closer to 1 to 10 ppm.

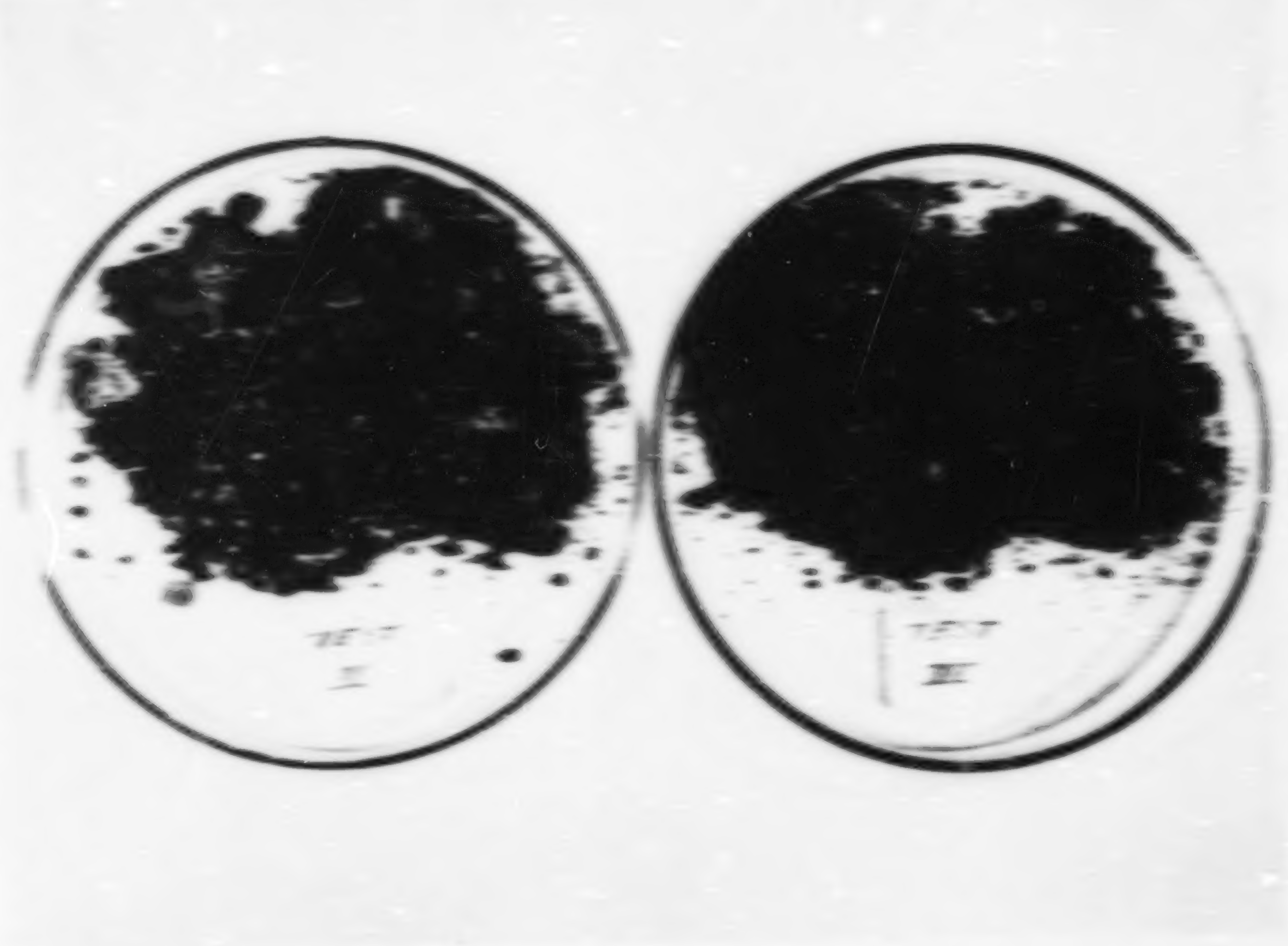


Figure 5-2. Photograph of solid residue samples.

TABLE 5-13. RESULTS OF TEST III SOLID RESIDUE ANALYSIS FOR PCBs BY GC/MS

| Compound | Estimated Concentration | |
|---|---|---|
| | mg PCB/kg Residue | Percent of Total PCB Compounds |
| Monochlorobiphenyl | ND[a] | - |
| Dichlorobiphenyl | 8 | 2 |
| Trichlorobiphenyl | 109 | 23 |
| Tetrachlorobiphenyl | 300 | 63 |
| Pentachlorobiphenyl | 52 | 11 |
| Hexachlorobiphenyl | 4 | 1 |

[a] ND = less than 0.1 mg/kg

Qualitative Survey Data. Evaporated aliquots of the solvent extracts were weighed, and the gravimetric results are shown in Table 5-14. Analysis by IR of the test sample residues from Test II found only silicone and ketone compounds. Major amounts of PCBs were found in the Test III samples. LRMS analysis of these samples confirmed the presence of PCBs in the Test III residue, as well as indicating their existence in the Test II residue. Since the LRMS method is more sensitive than the GC/MS analysis, this result denotes that PCBs are present in the Test II residue at levels below the GC/MS detection limits, or <0.1 ppm.

doped control sample was prepared to determine the recovery of PCBs from the solid residue material for the quantitative analyses. Thus this sample was not qualitatively analyzed beyond the gravimetric determination.

TABLE 5-14. SUMMARY OF SURVEY ANALYSIS OF SOLID RESIDUE EXTRACTS

| Test No. | Amount of Solids Extracted (kg) | Amount of Organic Residue in Extracts (mg) | Concentration of Extractables (mg/kg) |
|---|---|---|---|
| II-"Fluff" | 0.102 | 1.06 | 10 |
| III-Whole Capacitors | 0.111 | 44.7 | 404 |
| Doped Control[a] | 0.110 | 1.21 | - |

[a] Solid residue from Test II with 0.10 mg of Aroclor 1242 added.

## Inorganic Characterization

Approximately 100 mg aliquots of the solid residues from Tests II and III were completely dissolved for inorganic analysis. Major elements were determined within an accuracy factor of 100 percent by ICPOES, and a complete trace element survey was performed by SSMS within an accuracy factor of 500 percent. The results of the ICPOES and SSMS analyses are presented in Tables 5-14 and 5-15, respectively. Considering the relative accuracies of ICPOES and SSMS, the results of the two analyses agree generally well. The value for silicon determined by SSMS was lower than that determined by ICPOES. Silicon was probably lost as a volatile fluorosilicate during the preparation of the samples for the SSMS analysis.

The major elements composing the residues were found to be aluminum, iron, silicon, copper, tin, phosphorus, lead, zinc, and possibly potassium, all of which are the same constituents found to have predominated in samples from other areas of the kiln system.

TABLE 5-15. ANALYSIS OF SOLID RESIDUES BY ICPOES

| Element | Concentration (mg/g) |  | Element | Concentration (mg/g) |  |
|---|---|---|---|---|---|
|  | Test II | Test III |  | Test II | Test III |
| Al | 360 | 180 | Mg | 0.7 | 1 |
| Ag | 0.4 | 0.5 | Mn | 0.9 | 1 |
| As | ND(<0.1) | 0.9 | Mo | ≤0.6[a] | ≤0.5 |
| Ba | 0.2 | 1 | Ni | 0.3 | 0.4 |
| Be | ND(<0.001) | 0.002 | P | 17 | 13 |
| Ca | 3 | 6 | Pb | 7 | 4 |
| Cd | 0.04 | 0.07 | Si | 85 | 100 |
| Co | ≤0.1 | ≤0.1 | Sn | 13 | 7 |
| Cr | 0.7 | 0.7 | Sr | 0.01 | 0.02 |
| Cu | 50 | 43 | Ti | 0.9 | 1 |
| Fe | 300 | 400 | V | 0.1 | 0.1 |
| K | ≤180 | ≤160 | Zn | 2 | 5 |

a) The less than or equal to values indicate that the sample results were equal to or lower than the reagent blank.

## TABLE 5-16. ANALYSIS OF SOLID RESIDUES BY SSMS

| Element | Concentration (mg/g) |  | Element | Concentration (mg/g) |  |
|---|---|---|---|---|---|
|  | Test II | Test III |  | Test II | Test III |
| Pb | 4 | 3 | Cu | MC[b] | MC |
| Pt | 0.3 | 0.5 | Ni | ≤0.6 | ≤1 |
| W | ≤0.3[a] | ≤1 | Co | ≤0.02 | ≤0.09 |
| Pr | 0.02 | 0.02 | Fe | MC | MC |
| Ce | 0.06 | 0.05 | Mn | 2 | 1 |
| La | 0.06 | 0.05 | Cr | ≤1 | ≤0.09 |
| Ba | 0.3 | 3 | V | 0.1 | 0.09 |
| I | 0.02 | 0.01 | Ti | ≤1 | ≤1 |
| Sb | ≤0.01 | ≤0.07 | Sc | ≤0.004 | ≤0.007 |
| Sn | 5 | 3 | Ca | MC | MC |
| Cd | 0.1 | 0.09 | K | MC | MC |
| Ag | ≤0.08 | ≤1 | Cl | MC | MC |
| Mo | 0.07 | 0.1 | S | ≤1 | ≤0.9 |
| Zr | 0.1 | 0.2 | P | MC | MC |
| Y | 0.02 | 0.009 | Si | ≤1 | ≤0.9 |
| Sr | ≤0.07 | ≤0.06 | Al | MC | MC |
| Br | ≤0.09 | ≤0.4 | Mg | MC | MC |
| Se | 0.08 | 0.1 | F | MC | MC |
| As | ≤3 | ≤5 | Li | ≤0.02 | ≤0.004 |
| Ge | 0.008 | 0.03 | Ga | 0.2 | 0.1 |
| Z | ≤2 | ≤8 |  |  |  |

a) "≤", a less than or equal to sign indicates those elements which were detected but not significantly above background levels.

b) Major Constituent – present above quantifiable levels.

## 5.2 RESULTS OF NCB TESTS

### 5.2.1 Operating Conditions for Tests

Table 5-17 presents the operating conditions for the fuel oil background burn, R1(B), and the two test burns of NCB waste in diesel oil. More details of the operating conditions can be found in Appendix G. The major difference between Tests R3 and R4 was the temperature in the hot duct.

### 5.2.2 Destruction Efficiency and Composition of Combustion Zone Effluent Gas

Data on the composition of the combustion zone effluent were obtained from a variety of samples and types of analyses. The analyses, which included both quantitative and qualitative characterizations of the effluent and the analytical results, are described in detail in Appendix F. In this section of the report the analytical results are presented in a reduced form which facilitates interpretation of the data.

#### 5.2.2.1 Quantitative Characterization

The principal criteria for assessing the effectiveness of the incineration process for treatment of the waste were the calculated destruction efficiencies (DE's). The calculations of DE were based on the quantities of total organics and of nitrochlorobenzene found in the organic solvent extracts of the various hot zone sampling train components. Those data for the background test and two NCB tests are presented in Table 5-18. In estimating the total quantity of organic material collected, the estimates obtained by gas chromatographic analysis (of the unconcentrated extracts) and by gravimetric analysis (of an aliquot of concentrated extract evaporated to dryness) have been summed. This was done because other work in ADL laboratories has shown that many species with GC retention times similar to those found in these samples are lost by evaporation when an organic extract is dried to constant weight. As a result of this conservative method of estimation, the total organic emissions may be slightly overestimated.

In Table 5-19 the organic emission rates are expressed in terms of mg/cu m of hot zone effluent. These values are compared with the maximum organic loading that could have occurred if no destruction was accomplished. The latter quantity is simply the feed rate (waste and/or auxiliary fuel), in mg/min, divided by the total hot zone gaseous effluent flow, in standard cu m/min.

The calculated values of DE total and DE waste both suggest that destruction of the NCB waste was virtually complete in both tests. There was no decrease in the overall destruction efficiency (DE total) when the NCB waste was fed. The calculated destruction efficiency based only on NCB waste (DE waste) was within experimental error of 100%.

Other quantitative characterizations of the combustion zone effluent gas were provided by the on-line instruments, gas-detector tubes,

## TABLE 5-17. OPERATING CONDITIONS FOR TESTS ON NCB WASTES

| Test Number | Fuel Oil Background B1(B) | NCB Waste B3 | NCB Waste B4 |
|---|---|---|---|
| Hot Duct Temp. (°C) | 1074 | 1075 | 995 |
| Hot Duct Gas Flow s.cu.m/min (dry basis)(1) | 890 | 1000 | 1000 |
| Afterburner Temperature (°C) | 1288 | 1307 | 1332 |
| Fuel Oil/NCB Solution Rate to Loddby Burner (liters/hr) | 1780(2) | 2021 | 1764 |
| Fuel Oil Rate to Kiln (liters/hr) | 400 | 249 | 394 |
| Loddby Burner Flame Temp. (°C) | 1490 | 1548 | 1510 |
| Lime Consumption (32% $Ca(OH)_2$ by wt) (liters/hr) | 380 | 400 | 620 |
| Natural Gas (1000 cu.m./hr.) | 174 | 144 | 124 |
| Retention Time (seconds) | 2.6 | 2.3 | 2.3 |

(1) At 21°C

(2) Oil only

TABLE 5-18. SUMMARY OF QUANTITIES OF ORGANIC MATERIALS IN HOT ZONE EFFLUENT SAMPLE EXTRACTS

| Run | Sample ‡ | Estimated Total Organic Content, mg |  |  | Estimated NCB Content mg (by GC) |
|---|---|---|---|---|---|
|  |  | Total By GC | Total By Grav.* | Sum of GC and Grav. |  |
| R1(B) | PNF/CH$_2$Cl$_2$ | 19.4 | 48.4 | 67.8 | <0.2 |
|  | IT/CH$_2$Cl$_2$ | <1.0 | 6.8 | 7.8 | <0.02 |
|  | ST/Pentane | 14.4 | 23.8 | 38.2 | 0.06 |
|  | ST/Methanol | 1.8 | 97.4 | 99.2 | <0.01 |
|  |  | 36.6 | 176.4 | 213.0 | <0.3 |
| R3 | PNF/CH$_2$Cl$_2$ | 17.8 | 76.2 | 94.0 | <0.2 |
|  | IT/CH$_2$Cl$_2$ | <1.0 | 1.6 | 2.6 | <0.2 |
|  | ST/Pentane | 3.8 | 3.4 | 7.2 | 0.02 |
|  | ST/Methanol | 1.2 | 150.0 | 151.2 | <0.01 |
|  |  | 23.8 | 231.2 | 255.0 | <0.25 |
| R4 | PNF/CH$_2$Cl$_2$ | 13.0 | 53.2 | 66.2 | <0.2 |
|  | IT/CH$_2$Cl$_2$ | <1.0 | 47.2 | 48.2 | <0.02 |
|  | ST/Pentane | 23.5 | 9.0 | 32.5 | <0.01 |
|  | ST/Methanol | 0.4 | 53.8 | 54.2 | <0.01 |
|  |  | 37.9 | 163.2 | 201.0 | <0.24 |

‡ See Appendix F for sample codes.

* Based on residue remaining after evaporation of extract to dryness.

TABLE 5-19

HOT ZONE EMISSION RATES AND CALCULATED DESTRUCTION EFFICIENCIES

(see text for explanation)

| | R1(B) | R3 | R4 |
|---|---|---|---|
| Total Organics in HZ Effluent, mg/m$^3$ (PNF/$CH_2Cl_2$ plus IT/$CH_2Cl_2$ Plus ST/Pentane plus ST/Methanol) | 42.2 | 52.6 | 42.1 |
| Estimated NCB in HZ Effluent, mg/m$^3$ (PNF/$CH_2Cl_2$ plus IT/$CH_2Cl_2$ plus ST/Pentane plus ST/Methanol) | <0.06 | <0.05 | <0.05 |
| Calculated Maximum Total Organics in Effluent, if DE = 0, mg/m$^3$ | 26,500 | 33,400 | 31,500 |
| Calculated Maximum NCB in Effluent, if DE = 0, mg/m$^3$ | 0 | 8,350 | 7,290 |
| DE $_{Total}$ | 99.84 | 99.84 | 99.87 |
| DE $_{NCB\ Waste}$ | - | 99.99$_9$ | 99.99$_9$ |

and analysis of the hot zone train impinger solutions.* Data obtained from the on-line instruments and gas-detector tubes are presented in Table 5-20.

The "not detectable" notation for volatile hydrocarbons means that no difference in signal intensity could be observed between the sample gas and cylinder "zero" air.

Because the gas-detector tubes indicated very low levels of chlorine phosgene and sulfur dioxide, no more precise quantitative methods were applied for these species.

It is unfortunate that the chemiluminescent analyzer appeared to be unable to detect nitrogen dioxide during these tests (see Section 4.2.5.3 ). Calculations based on the nitrogen content of the NCB waste and total hot zone effluent flow rates suggest that about 2500 ppm of nitrogen dioxide is the theoretical maximum yield. The data obtained during field testing do not allow determination of the actual nitrogen dioxide concentration in the combustion zone effluent. The apparuntly low level of hydrochloric acid in the gas-detector tube assay is probably due to scrubbing of the acid by droplets of water that condensed in the sampling line after it left the hydrocarbon analyzer.

The hydrochloric acid content of the combustion zone effluent was also estimated by analysis of the dry impinger and sodium acetate impinger components of each hot zone sample. The estimates thus obtained for chloride content of the gaseous effluent were**:

| | |
|---|---|
| R1(B) | 0.51 g/m$^3$ |
| R3 | 5.26 g/m$^3$ |
| R4 | 6.96 g/m$^3$ |

The calculated maximum theoretical loadings of hydrochloric acid, based on the feed rate and elemental analyses were:

R1 (B) = 0.23 g/m$^3$
R3 = 3.02 g/m$^3$
R4 = 2.67 g/m$^3$

Except for the background tests, the actual estimated chloride loadings were much higher than the calculated maxima. It does not seem probable that the discrepancy is due to systematic errors in the analysis. The most that can

---

* The gas bulb samples were not useful. See Appendix F.

** Value stated is a sum of data from dry impinger plus sodium acetate impingers for each run; $Cl^-$ in g/m$^3$ = ($Cl^-$ in ppm x total volume of solution in l) ÷ (total volume sampled in m$^3$ x 1000).

## TABLE 5-20. QUANTITATIVE ANALYSIS RESULTS OBTAINED FROM ON-LINE INSTRUMENTS AND GAS-DETECTING TUBES

| | R1(B) | R3 | R4 |
|---|---|---|---|
| | Concentration (volume basis)* | | |
| On-Line Instruments | | | |
| Volatile Hydrocarbons | N.D.†<br><5 ppm | N.D.†<br><5 ppm | N.D.†<br><5 ppm |
| Carbon Monoxide | 7 ppm | 7 ppm | 8 ppm |
| Carbon Dioxide | 7.3% | 7.0% | 6.7% |
| Oxygen** | 9.4% | 9.7% | 10.0% |
| Nitrous Oxide (NO) | 50 ppm | 170 ppm | 165 ppm |
| Gas-Detector Tubes | | | |
| Chlorine | N.D.†<br><1 ppm | N.D.†<br><1 ppm | N.D.†<br><1 ppm |
| Hydrochloric Acid | N.D.†<br><2 ppm | N.D.†<br><2 ppm | N.D.†<br><2 ppm |
| Phosgene | N.D.†<br><1 ppm | N.D.†<br><1 ppm | N.D.†<br><1 ppm |
| Sulfur Dioxide | <10 ppm | 12 ppm | 15 ppm |
| Nitrogen Dioxide †† | > | >300 ppm | >300 ppm |

* Wet gas basis

** Estimated from $CO_2$ concentration for R3 and R4

† Not detectable

†† High range tubes were not available on-site when the $NO_x$ analyzer failed to detect $NO_2$

be said, based on the available data, is that observed hydrochloric acid hot zone emissions agree with expected emissions to within a factor of two or three.

### 5.2.2.2 Qualitative Characterization

Qualitative characterization of the organic material found in the solvent extracts of the various hot zone sampling train components is somewhat less straightforward than the quantitative treatment above. It should be noted at the outset, however, that none of the organic species identified in the various effluent samples appear to be especially hazardous. The only chlorinated hydrocarbon, for example, found in any of the samples was a species with a molecular weight of 200 and an empirical formula of $C_{10}H_{13}O_2Cl$, in the R3 sorbent trap pentane extract; the total quantity found corresponded to about 0.3 mg/m$^3$.

The organic chemical species which were identified in samples but not present in the blanks are listed in Table 5-21. Estimated upper limits on concentration are given for species which may be present at >.1 mg/m$^3$. The concentrations shown are almost certainly overestimates in all cases; they were calculated by assuming that the relative abundances indicated by LRMS were applicable to the entire sample and by assuming that the total sample weight was the sum of the GC and gravimetric estimates. The compounds found can be grouped into several categories:

- The first four species are normally expected products of combustion of aromatic fuels (#2 diesel oil or NCB waste) and thus not of particular concern.

- The presence of dibutyl tin dichloride probably indicates that the purge time allowed before each test was insufficient to eliminate all residues from the wastes burned during the previous shift. The organo tin species is not a component of either the diesel oil or the NCB waste. Tin, in any form, is not present in the feed in sufficient quantity to account for this species in the effluent. It is interesting to note that the dibutyl tin dichloride was found primarily in the pentane extract from the sorbent trap.

- The "nitrogen-containing compounds" which are indicated as present in substantial quantities in the R3 and R4 hot zone samples are high molecular weight species of unknown structure. Their presence in the methanol extracts of the two NCB test sorbent traps was inferred from the LRMS spectra. Fairly low molecular weight nitrogenous fragments appeared in the spectra at probe temperatures (>300°C) much higher than would be consistent with their molecular weights.

  It is possible that these compounds are contaminants, but since they do not appear in the blank or in the background test sample, it seems most reasonable to presume that they

TABLE 5-21. ORGANIC CHEMICAL SPECIES FOUND IN HOT ZONE EFFLUENT SAMPLE EXTRACTS*

|  | Approximate Concentration, mg/m$^3$ |  |  |
|---|---|---|---|
| Species | R1(B) | R3 | R4 |
| Benzoic Acid | 1.8 | 1.3 | n.f.† |
| Alkyl Benzenes | n.f. | <1.0 | n.f. |
| Toluic Acid | n.f. | <0.1 | n.f. |
| Ethyl Benzoate | n.f. | <0.1 | n.f. |
| Dibutyl Tin Dichloride | 1.1 | <1.0 | 3.1 |
| Nitrogen-Containing Compounds | n.f. | 10.0 | 6.0 |
| Silicones | 3.0 | <0.1 | 3.9 |
| MW 326 (Hydroxy Octoxy Benzophenone?) | n.f. | n.f. | 10.0 |
| MW 346 (Oxygenated) | n.f. | n.f. | 2.0 |
| Nonyl Phenol | n.f. | n.f. | <1.0 |
| Phenol | n.f. | <0.1 | n.f. |
| $C_{10}H_{13}O_2Cl$, MW 200 | n.f. | <0.1 | n.f. |
| Dihydrofuran | <1.0 | n.f. | n.f. |
| Furfural | <1.0 | n.f. | n.f. |
| m/e 530 | n.f. | n.f. | <1.0 |

* Sum of quantities extracted from probe wash, filter, dry impinger and sorbent trap for each run. Quantitative estimates are based on LRMS abundance data (Appendix F) and total organic emissions (Table 5-2).

† Not found.

arise from some degradation of the NCB waste. These species are not chlorinated and are not highly aromatic. The possible hazard associated with these species cannot be assessed, but the types of compounds implied by the available analytical data are not among those presently known to be especially hazardous.

- The silicones and the MW 326 species, tentatively identified as hydroxy octoxy benzophenone, are probably contaminants. The latter species is found in some of the blanks (the mixed solvent used as a dry impinger blank and the ST/methanol blank) as well as in some sample extracts. The MW 346 compound found in the R4 probe wash and filter extract is probably also a contaminant.

- The various oxygenated species found at concentrations of <1 mg/m$^3$ in all three tests are probably combustion products. None appears to be present at high enough concentration to cause serious concern.

The conclusion to be drawn from these results is that the organic species identified in the hot zone effluent during the NCB tests do not include any compounds that are known to be hazardous at the concentrations found in these tests. The only species identified in the hot zone effluent which would clearly require some form of emission control are hydrochloric acid and nitrogen oxides.

Although the nitrate concentration in the sodium acetate impingers was measured, this medium was not selected for this purpose and is not well suited for trapping of nitrogen oxides. The data obtained indicated little difference between the background and the waste tests, which implies that trapping efficiency was indeed low. The calculated maximum yield* of nitrogen oxides, as $NO_2$, is 2.46 g/m$^3$ for R3 and 2.14 g/m$^3$ for R4. In the absence of reliable data, it must be presumed that actual levels approached these maxima.

### 5.2.3 Final Emissions

### 5.2.3.1 Stack Gas

The total particulate loading as measured by EPA Method 5 was:

| | |
|---|---|
| R1(B) | 38.1 mg/m$^3$ |
| R3 | 13.9 mg/m$^3$ |
| R4 | 16.4 mg/m$^3$ |

The estimated emissions of hydrochloric acid, based on analysis of the sampling train impingers, were:

---

* Assuming all nitrogen in the waste was converted to nitrogen dioxide.

| | |
|---|---|
| R1(B) | 2.5 mg/m$^3$ as $Cl^-$ |
| R3 | 12.5 mg/m$^3$ |
| R4 | 13.0 mg/m$^3$ |

These data, when compared to the chloride levels estimated for the hot zone effluent, imply that the scrubber efficiency is excellent. Calculated chloride removal efficiencies are 99.5% for R1(B) and 99.8% for R3 and R4.

No other species were sought in the stack train samples.

### 5.2.3.2 Scrubber Water

There was no evidence from gas chromatography, IR spectra, or LRMS analyses of the methylene chloride extracts of the scrubber water samples that any significant quantity of organic material was present. The total quantity of material (GC estimate plus gravimetric determination) was 2.0 mg/liter for the fresh scrubber water and <1.4 mg/liter for the background test spent scrubber water. For the two waste tests, the spent scrubber water levels were estimated as <1 mg/liter (R3) and <1.4 mg/liter (R4). No particular organic species were identified for these samples because the total quantities were so low.

The scrubber water samples were also analyzed for chloride and nitrate content. The data are presented in Table 5-22. The chloride data are in good agreement with the estimates obtained from analyses of the hot zone sampling train impingers. This confirms the implication of the stack impinger data that the scrubber efficiency for removal of HCl is high (see data above). The nitrate data indicate that the scrubber did not remove substantial quantities of nitrogen oxides from the combustion zone effluent.

## TABLE 5-22. CHLORIDE AND NITRATE ANALYSES OF SCRUBBER WASTES

|  | $Cl^-$<br>mg/liter | $NO_3^-$<br>mg/liter |
|---|---|---|
| Fresh Scrubber Water |  |  |
| R0 - S1 | 24 | 0.3 |
| Spent Scrubber Water |  |  |
| R1(B) - S0 | 208 | 47.8 |
| R3 - S0 | 1750* | 67.2 |
| R4 - S0 | 1815* | 63.5 |

*These values correspond to estimated concentrations of 5.94 g/m$^3$ (R3) and 6.16 g/m$^3$ (R4) of chloride in the incinerator effluent gas. Analysis of the hot zone impinger solutions implied 5.26 g/m$^3$ and 6.96 g/m$^3$, respectively, which is in very good agreement with the scrubber water assays.

# 6. WASTE INCINERATION COST

Individual economic analyses were performed to determine the commercial (i.e., contract off-site) and industrial costs of incinerating the used PCB-containing capacitor and NCB process wastes tested at the Rollins Environmental Service Incorporated incinerator near Houston, Texas. The economic analyses were divided into capital investment and annual operating costs. For the commercial disposal facilities (one to be used exclusively for used capacitor wastes and one to be used four months per year for NCB process wastes), equipment prices, fuel consumption, and manpower requirements estimates for the conveyor fed venturi scrubber-equipped incinerator system were based on data obtained from Rollins Environmental Services Incorporated. For the on-site industrial facility for disposal of NCB process wastes, equipment prices, fuel consumption, and manpower requirements estimates were based on engineering scaling of data obtained for the Chemolite plant incinerator system from the 3M Company. The costs of other portions of the disposal facilities and associated labor were estimated using the method of Happel ("Chemical Process Economics," second edition, John Happel and Donald G. Jordan, 1974), data from Guthrie ("Capital Cost Estimating," Chemical Engineering, March 24, 1969), and standard engineering reference methods. Equipment costs were adjusted to March 1976 prices using the Engineering News Record Index. Land prices are not included in any of the disposal plant cost estimates. Transportation costs were included for the economic analyses of commercial facilities for disposal of used capacitor and NCB process wastes, which were premised upon central facilities at Houston, Texas, incinerating waste materials transported 850 kilometers (500 miles). Transportation costs were not included for the economic analyses of the on-site industrial facility for disposal of NCB process wastes, since the incinerator was assumed to be located at the chemical plant generating the waste to be disposed.

## 6.1 CAPITAL INVESTMENT

The capital investment for the facility to incinerate 5,000 metric tons per year of used PCB-containing capacitors, as shown in Table 6-1, is based upon a design concept which employs an enclosed one-metric-ton-per-hour hammermill to mill the used capacitors, an enclosed conveyor belt feed, a 3.7 meter diameter solid waste kiln, and a 1.6 meter diameter Loddby liquid waste burner exhausting into a 10.6 meter long afterburner. The system is equipped with a venturi scrubber, Flexitray absorber, demister, and stack system. The facility costs include exhaust of the enclosed hammermill and conveyor to the incinerator, induced draft fans (600 KW total), fuel oil storage tank farm (one-month capacity), lime slurry tank, fuel oil feed pumps, 30.5 meter stack, and stabilization lagoon system for disposal of neutralized lime slurry/scrubber wastewater discharge. It was assumed that fuel oil and natural gas consumption rates per kilogram of milled capacitor

TABLE 6-1. CAPITAL INVESTMENT

5000 METRIC TONS/YEAR* USED PCB CAPACITOR WASTE INCINERATION PLANT

| Equipment | Size | Estimated Costs** Equipment# | Labor | |
|---|---|---|---|---|
| 1-Venturi Scrubber, absorber, pumps, and stack | 5,300 lpm | $ 88,000 | | |
| 2-Induced draft fans, including motors | 300 HP each | 69,000 | | |
| 2-Feed pumps | 1,300 lpm each | 4,600 | | |
| 1-Tank farm, including lime slurry tank | 1,900 cu.m. | 84,400 | | |
| 1-Hammermill, enclosed, fan and ducting | 0.9 metric tons/hr. | 9,900 | | |
| | | $255,900 | $ 25,600 | |
| Instruments (10% of equipment) | | 25,600 | 3,800 | |
| (Key Accounts) | | $281,500 | $ 29,400 | |
| Insulation (10% of key accounts) | | 28,200 | 42,200 | |
| Piping (45% of key accounts) | | 126,700 | 126,700 | |
| Foundations ( 4% of key accounts) | | 11,300 | 16,900 | |
| Buildings ( 4% of key accounts) | | 11,300 | 7,500 | |
| Structures ( 4% of key accounts) | | 11,300 | 2,300 | |
| Fire Protection (0.75% of key accounts) | | 2,100 | 13,700 | |
| Electrical (4.5% of key accounts) | | 12,700 | 19,000 | |
| Painting and Clean-up (0.75% of key accounts) | | 2,100 | 13,700 | |
| Subtotal | | $487,200 | $271,800 | $ 759,000 |
| Installed Costs of Special Equipment: | | | | |
| 1-Incinerator, conveyor fed | 24 million Kcal/hr. | | | 1,500,000 |
| 1-Stabilization lagoon system | 30,000 cu.m. | | | 80,000 |
| Equipment and Labor | | | | $2,339,000 |
| Overheads (30% of Equipment & Labor) | | | | 701,700 |
| Total Erected Cost | | | | $3,040,700 |
| Engineering Fee (10% of Erected Cost) | | | | 304,100 |
| Contingency (10% of Erected Cost) | | | | 304,100 |
| Total Capital Investment | | | | $3,648,900 |

* 16 metric tons/day

** Mar. 1976 basis

# F.O.B. cost

waste were 3.6 liters and 136.7 cubic meters, respectively, based on extrapolation of test results to maximum waste feed rate by Rollins Environmental Services Incorporated.

The size of the facility was based upon three-shift, seven-day per week, 52-week per year operation to dispose of 5,000 metric tons of used PCB-containing capacitors. Annual production of medium and small sized PCB-containing capacitors had been approximately 45,400 metric tons per year through 1976, based on use of 11,340 metric tons of PCB per year for this purpose, at an average PCB weight content of 25 percent. The used capacitor incineration plant has a nominal thermal capacity of 24 million Kcal per hour; the actual thermal load is estimated at 94 percent of nominal capacity, based on 2411 liters per hour of No. 2 fuel oil and 90.6 cubic meters per hour of natural gas. Heating value of the milled capacitor waste is less than 3,000 Kcal per kilogram. The total capital investment for the used capacitor waste facility is estimated at $3,648,900.

The total capital investment shown in Table 6-2 for the contract disposal facility required to incinerate 4,540 metric tons of nitrochlorobenzene process wastes in four months is based upon the foregoing Rollins Environmental Service incinerator used to burn a blend of thermally equivalent quantities of No. 2 fuel oil and NCB wastes at the rate of 18,100,000 Kcal per hour. Additional waste storage tankage, to store 1140 cubic meters of NCB process wastes, has been included in the capital outlay estimate. The total capital outlay of $3,749,800 shown for the central facility has been divided pro-rata to an allocation of $1,249,900 for contract NCB waste incineration, since the facility spends only one-third of its time in this service.

Since the estimated total annual quantities of approximately 9100 metric tons of NCB process wastes for the United States appear to be large enough to support an on-site incinerator installation in addition to the contract facility, capital cost estimates for an on-site plant have been developed. It was assumed, for incineration at the generating site, that less auxiliary fuel would be used (30 percent of the total heat input), and that a smaller, continuous service incinerator would suffice. Consequently, the capital estimate of $2,016,000 shown in Table 6-3 has been based on engineering scale-down of the 22.7 million Kcal per hour incinerator used by the 3M Company at their Chemolite plant to the 4.3 million Kcal per hour incinerator system required for continuous on-site NCB waste disposal.

## 6.2 ANNUAL OPERATING COSTS

The annual operating costs for the commercial and industrial facilities consist of labor, fuel, other utility, hydrated lime and freight costs (where applicable), plus cost of capital, equipment depreciation, maintenance, taxes and insurance. The labor costs for the contract disposal facilities located near Houston, Texas, have been calculated on the number of personnel assigned to operate the system at the rates given by Rollins Environmental Services Incorporated. The labor costs for the on-site NCB incineration facility have been calculated on the basis of data from the 3M Company. Costs for supervision, supplies, and payroll-related expense have been included for all facilities at rates prevalent in the chemical industry.

TABLE 6-2. CAPITAL INVESTMENT

CENTRAL FACILITY FOR INCINERATION - 4540 METRIC TONS/4-MONTH PERIOD NCB WASTE

| Equipment | Size | Estimated Costs* Equipment** | Estimated Costs* Labor | |
|---|---|---|---|---|
| 1-Venturi scrubber, absorber, pumps and stack | 5,300 Tpm | $ 88,000 | | |
| 2-Induced draft fans, including motors | 300 KW each | 68,000 | | |
| 2-Feed pumps | 1,300 Tph each | 4,600 | | |
| 1-Tank farm, including lime slurry tank | 3,040 cu.m. | 116,200 | | |
| | | $277,800 | $ 27,800 | |
| Instruments (10% of equipment) | | 27,800 | 4,250 | |
| (Key Accounts) | | $305,600 | $ 32,300 | |
| Insulation (10% of key accounts) | | 30,600 | 46,000 | |
| Piping (45% of key accounts) | | 137,500 | 137,500 | |
| Foundations ( 4% of key accounts) | | 12,200 | 18,300 | |
| Buildings ( 4% of key accounts) | | 12,200 | 8,600 | |
| Structures ( 4% of key accounts) | | 12,200 | 2,400 | |
| Fire Protection (0.75% of key accounts) | | 2,300 | 14,900 | |
| Electrical (4.5% of key accounts) | | 13,800 | 25,600 | |
| Painting and Clean-up (0.75% of key accounts) | | 2,300 | 14,900 | |
| Subtotal | | $528,700 | $295,000 | $ 823,700 |
| Installed Costs of Special Equipment: | | | | |
| 1-Incinerator, conveyor fed | 24 million Kcal/hr. | | | 1,500,000 |
| 1-Stabilization lagoon system | 30,000 cu.m. | | | 80,000 |
| Equipment and Labor | | | | $2,403,700 |
| Overheads (30% of Equipment & Labor) | | | | 721,100 |
| Total Erected Cost | | | | $3,124,800 |
| Engineering Fee (10% of Erected Cost) | | | | 312,500 |
| Contingency (10% of Erected Cost) | | | | 312,500 |
| Total Capital Investment | | | | $3,749,800 |
| Pro-rata Total Capital Investment for NCB Waste Incineration | | | | $1,249,900 |

* Mar. 1976 basis

** F.O.B. Cost

## TABLE 6-3. CAPITAL INVESTMENT

## 4540 METRIC TONS/YEAR NGB WASTE INCINERATION PLANT (ON-SITE)

| Equipment | Size | Estimated Costs<br>Equipment | <br>Labor | |
|---|---|---|---|---|
| 1-Scrubber, absorber, pumps and stack | 1,700 lpm | $ 98,200 | | |
| 3-Induced draft fans | 75 KW, total | 45,500 | | |
| 1-Well water supply system | 1,700 lpm | 21,200 | | |
| 1-Waste water treatment system | 1,700 lpm | 72,700 | | |
| 1-Incinerator system, including feed system | 4.3 million Kcal/hr. | 318,200 | | |
| | | $555,800 | $ 46,800 | |
| Instruments (10% of equipment) | | 55,600 | 8,300 | |
| (Key Accounts) | | $611,400 | $ 55,100 | |
| Insulation (10% of key accounts) | | 61,100 | 91,700 | |
| Piping (45% of key accounts) | | 275,100 | 275,100 | |
| Foundations ( 4% of key accounts) | | 24,500 | 36,700 | |
| Buildings ( 4% of key accounts) | | 24,500 | 17,100 | |
| Structures ( 4% of key accounts) | | 24,500 | 4,500 | |
| Fire Protection (0.75% of key accounts) | | 4,600 | 29,800 | |
| Electrical (4.5% of key accounts) | | 27,500 | 47,300 | |
| Painting and Clean-up (0.75% of key accounts) | | 4,600 | 29,600 | |
| Subtotal | | $1,057,800 | $590,300 | $1,648,100 |
| Installed Costs of Special Equipment | | | | |
| Refractories | | | | 157,600 |
| Equipment and Labor | | | | $1,805,700 |
| Overheads (30% of Equipment & Labor) | | | | 541,700 |
| Total Erected Cost | | | | $2,347,400 |
| Engineering Fee (10% of Erected Cost) | | | | 234,700 |
| Contingency (10% of Erected Cost) | | | | 234,700 |
| Total Capital Investment | | | | $2,816,800 |

The utility costs include those for electricity. Water costs, based on actual consumption data, have been included only for the on-site NCB incinerator, since the Houston, Texas, located contract disposal facility has its own well-water system and its water costs were covered under electric power costs. The amount of No. 2 fuel oil consumed was based on extrapolation of actual test data for the two wastes at the Rollins Environmental Services, Deer Park, Texas, site.

The annual operating costs for the contract incineration of 5,000 metric tons per year of used PCB-containing capacitors are summarized in Table 6-4. The estimated annual operating expense for the plant, based on 21-shift per week operation, is $3,704,600 or $740.92 per metric ton. Almost $307 of this very high disposal cost per metric ton is cost of the fuel oil and natural gas required to yield the temperatures needed for total incineration of the PCB, since the waste itself has no appreciable heating value. Disposal costs would be significantly reduced if a high heating value liquid waste could be utilized in place of fuel oil to support the incineration of the PCB.

The annual operating costs for the contract disposal and on-site plants for incineration of NCB wastes are shown in Tables 6-5 and 6-6, respectively. For contract disposal, using the pro-rata capital investment value of $1,249,900 given in Table 6-2, and four months of 21-shift per week operation on NCB process wastes, the estimated total operating cost is $1,099,200, equivalent to $242.11 per metric ton. The total annual corresponding operating cost for the disposal of the same tonnage (4540 metric tons) of NCB waste on-site, given in Table 6-5 is $1,283,300, equivalent to $282.67 per metric ton. This figure is also based on 21-shift per week operation.

The cost of capital shown is based on the assumption that private debt financing is used for each facility.

## TABLE 6-4. ANNUAL OPERATING COST

## 5000 METRIC TONS/YEAR* USED PCB CAPACITOR WASTE INCINERATION PLANT

| Item | | Cost - $/Year |
|---|---|---|
| Depreciation (15% of plant investment) | | $ 547,300 |
| Cost of Capital (10% of plant investment) | | 364,900 |
| Maintenance (9% of plant investment) | | 328,400 |
| Utilities | | 1,675,420 |
| Electric power [(252 kW (24)(310)] $0.075 | = $ 140,620 | |
| Water [3270 lpm (1440) (310) @ $0.008/1000 liter | = — ** | |
| Fuel Oil, No. 2, 114,400 bbl @ $13.00/bbl | = 1,487,200 | |
| Natural Gas [90.6 cu.m./hr (24)(310)] @ $70.63/1000 cu.m. | = 47,600 | |
| Solid Waste Disposal | | — *** |
| Freight (500 mi. @ $1.71/cwt) 5,000 metric tons @ $37.70/metric ton | | 188,500 |
| Chemicals | | |
| Hydrated Lime, 3,750 metric tons @ $35.00/metric ton | | 131,300 |
| Labor | | 395,800 |
| Chief Operator - 1 x 24 x 260 x $8.05 | = 50,200 | |
| Operators - 3 x 24 x 365 x $5.95 | = 102,600 | |
| Supervision (15% of Operating Labor) | = 38,900 | |
| Supplies (20% of Operating Labor) | = 46,600 | |
| Payroll Related Expense (35% of Operating Labor) | = 87,500 | |
| Taxes and Insurance (2% of plant investment) | | 73,000 |
| | Total | $3,704,600 |

Cost per metric ton of used PCB capacitor waste @ 5,000 metric tons/year $ 740.92

* 16 metric tons/day
** From wells—cost included in Total Capital Investment and Electric Power Costs
*** Offset by aluminum recovery

## TABLE 6-5. PRO-RATA ANNUAL OPERATING COST

## CENTRAL FACILITY FOR INCINERATION-4540 METRIC TONS/4-MONTH PERIOD HCB WASTE

| Item | | Cost - $/4 Month Period |
|---|---|---|
| Depreciation (15% of pro-rata plant investment) | | $ 187,500 |
| Cost of Capital (10% of pro-rata plant investment) | | 125,000 |
| Maintenance (9% of pro-rata plant investment) | | 112,500 |
| Utilities | | 292,100 |
| Electric power [(1260 kW (24)(104)] $0.075 | = $ 47,200 | |
| Water [(4214 ton (1440)(104)] @ $0.006/1000 ] | = —— * | |
| Fuel Oil, No. 2, 16,490 bbl @ $13.10/bbl | = 274,400 | |
| Natural Gas [(174.6 cu.m./hr)(24)(104) @ $70.63/1000 cu.m. | = 20,200 | |
| Freight (500 mi. @ $7.77/cwt) 4540 metric tons @ $37.70/metric ton | | 171,200 |
| Chemicals | | |
| Hydrated lime, 1,817 metric tons @ $35.00/metric ton | | 63,600 |
| Labor | | 132,300 |
| Chief Operator - 1 x 24 x 104 x $6.75 | = 16,800 | |
| Operators - 3 x 24 x 104 x $8.18 | = 61,200 | |
| Supervision (15% of Operating Labor) | = 11,700 | |
| Supplies (20% of Operating Labor) | = 15,600 | |
| Payroll Related Expense (35% of Operating Labor) | = 27,200 | |
| Taxes and Insurance (2% of pro-rata plant investment) | | 25,000 |
| | Total | $1,099,200 |

| | |
|---|---|
| Cost per metric ton of HCB waste | $ 242.11 |

TABLE 6-6. ANNUAL OPERATING COST

4540 METRIC TONS/YEAR NCB WASTE INCINERATION PLANT (ON-SITE)

| Item | | Cost - $/Year |
|---|---|---|
| Depreciation (15% of plant investment) | | $ 422,500 |
| Cost of Capital (10% of plant investment) | | 281,700 |
| Maintenance (5% of plant investment) | | 140,800 |
| Utilities | | 110,000 |
| Electric power [120 kW (24)(330)] $0.015 | = $ 14,300 | |
| Water [1100 lpm (1440)(330)] @ $0.008/1000 l | = 4,200 | |
| Fuel Oil, No. 2, 7040 bbl @ $13.00/bbl | = 91,500 | |
| Chemicals | | |
| Hydrated lime, 1,077 metric tons | | 63,600 |
| Labor | | 208,400 |
| Operators - 2 x 24 x 365 x $7.00 | = 122,600 | |
| Supervision (15% of Operating Labor) | = 18,400 | |
| Supplies (20% of Operating Labor) | = 24,500 | |
| Payroll Related Expense (35% of Operating Labor) | = 42,900 | |
| Taxes and Insurance (2% of plant investment) | | 56,300 |
| | Total | $1,283,300 |

Cost per metric ton of NCB waste @ 4540 metric tons/year $ 282.67

## 7. REFERENCES

1. TRW Document #27033-6005-RU-00, "Analytical Plan for Facility No. 8 Tests to be Conducted at Rollins Environmental Services, Inc.," November 1976.

2. "Destroying Chemical Wastes in Commercial Scale Incinerators Facility Report No. 3, Systems Technology," published under EPA Contract No. 68-01-2966, November 1976.

3. Environmental Protection Agency, Standards of Performance for New Stationary Sources, Federal Register, Vol. 41, No. 111, June 8, 1976.

## APPENDIX A

# ASSESSMENT OF ENVIRONMENTAL IMPACT OF DESTROYING CHEMICAL WASTES
# AT
# ROLLINS ENVIRONMENTAL SERVICES, INC.
# DEER PARK, TEXAS*

The Rollins Deer Park facility is located about 20 miles east of Houston in a highly industrialized area near the Houston ship channel. This rotary kiln incinerator will be evaluated for its capability of thermal destruction of the following industrial chemical wastes:

1) Nitrochlorobenzene - 7,000 gallons

2) PCB wastes in capacitors - 4,000 lbs whole capacitors
10,000 lbs hammermilled capacitors

State and federal permits have been issued to Rollins authorizing both air and water emissions, including:

Texas Air Control Board, Permit No. R-679

Texas Water Quality Board, Permit No. 01429

National Pollutant Discharge Elimination System, Permit No. TX0005941

The Rollins incineration system consists of a rotary kiln and a liquid injection burner feeding into a common afterburner. Total heat release is 110 million Btu/hr. Solid wastes are conveyor fed into the rotary kiln and reacted at temperatures from 1200 to 1500°F. Combustion gases are passed through an afterburner, fired by a liquid injection burner, to attain temperatures of 2300 to 2400°F. Estimated total residence time is 2.6 seconds. Ash from the kiln is disposed of on-site in an approved landfill.

A venturi scrubber is used to remove particulate from the exhaust gases. Lime is injected to neutralize the scrubber water in a single-pass system. The used scrubber water then enters settling ponds and is further treated, if required, before eventually entering the Houston ship channel. Exhaust gases also pass through absorption trays and a mist eliminator before entering the stack. The exhaust stack is 100 feet high with a sampling platform and ports about 55 ft above ground level. Standard sample ports 90° apart are provided. The exhaust plume was white with slight fallout on the day of the site visit.

Operating at this site since 1971, Rollins incinerates approximately 250,000 lbs of waste per day, consisting of bulk liquids, solids, and slurries. The facility operates 24 hours a day, 7 days a week, and has 42 employees. About 10-15 trucks enter the site for unloading each day. Routine maintenance is performed four times a year. The facility is relatively clean and odor-free for a commercial destruction facility.

Numerous refineries and chemical plants surround the Rollins facility. There are grass, brush, and small trees in the area. Local wildlife include quail, dove, rabbits, and fox. There are some cattle grazing in the area. The nearest residences are 3 to 4 miles from the site. Prevailing winds are 10-12 miles per hour from the southeast.

A maximum of 600,000 gallons of waste are stored on-site for period averaging two weeks. A spill prevention plan is in effect. Wastes are analyzed prior to incineration by an on-site laboratory. Operation of the incinerator is not considered to be noisy.

The potential detrimental environmental effects are expected to result from: 1) storage and handling of wastes prior to destruction, 2) emissions occurring during tests, and 3) disposal of liquid and solid residue remaining after combustion. The most significant hazard would result from contact with waste liquid and/or fumes during a spill.

The nitrochlorobenzene waste is a brownish liquid containing 90% isomers of nitrochlorobenzene. The p-nitrochlorobenzene, the desired product at the waste generation source and probably also the major constituent of the nitrochlorobenzene waste, is a significant toxic hazard. The 1973 Toxic Substances List provides the following data on p-nitrochlorobenzene and m-nitrochlorobenzene:

p-nitrochlorobenzene

Oral Toxicity: $LD_{50}$ (rat) = 420 mg/kg

Dermal Toxicity: OSHA (skin) = 1 $mg/M^3$

m-nitrochlorobenzene

Oral Toxicity: $LD_{50}$ (rat) = 2460 mg/kg

In addition, the mixture has a flash point of 264°F. The waste m- and o-nitrochlorobenzenes are volatile solids which give off flammable vapors when heated and may form explosive mixtures with air. The waste is also incompatible with strong oxidizing agents and should not be stored near strong oxidizing agents. Since the nitrochlorobenzenes are also toxic by inhalation, inhalation of the vapors should be avoided at all times. In the case of skin contact, the skin should be washed off immediately with water and soap. The effects of overexposure to the nitrochlorobenzene waste include blue tint to fingernails, lips and ears indicative of cyanosis, and headache, drowsiness and nausea followed by unconsciousness.

The capacitor wastes contain membranes impregnated with PCB. The general environmental contamination of PCB, resulting in adverse effects on certain forms of animal life, is well known. The studies conducted so far have indicated that some persons carry a body burden of PCB in their fat tissue. In addition, PCBs have been shown to accumulate in fish and aquatic invertebrates to levels of 75,000 times that present in water. Thus PCB is definitely bioaccumulative. The 1973 Toxic Substances List provides the following data on the type of PCB used in capacitors:

Aroclor 1242 [Chlorobiphenyl (42% Cl)]

Inhalation Toxicity: Toxic Concentration (human) - 10 mg/M$^3$

Dermal Toxicity: Lethal Dose (rabbit) - 794 mg/kg

OSHA (skin) - 1 mg/M$^3$

Oral Toxicity: LD$_{50}$ (rat) - 8650 mg/kg

### Storage and Handling

Liquid wastes will be received by tank truck and transferred to storage/run tanks. Solid wastes will be received by truck and stored in the original containers until incinerated. Leaks and any spills will be washed down with water or absorbed. All rinse or wash down liquids, or absorbent, will be incinerated, or neutralized prior to disposal.

### Incineration Tests

Operating temperature and residence time of the incinerator and afterburner should provide essentially complete combustion of the wastes, resulting in harmless exhaust emissions. On-line monitoring of gases from the combustion zone will be utilized as an indication of combustion efficiency. In addition, stack emissions (downstream of scrubber) will be checked for hazardous gaseous species using Gastec® analyzer tubes for specific gases and vapors.

### Disposal of Residues

Residue material from the incineration process will consist of scrubber water and ash. Liquid residue from the scrubber will be analyzed by Rollins personnel before discharge to the channel. Solid residues (ash) will also be tested prior to on-site landfill. Wastes remaining in the storage/run tanks and residual solids will be incinerated at the conclusion of the test program.

# APPENDIX B

# SAMPLE VOLUME DATA - PCB TESTS

## TABLE B-1. SAMPLING SYSTEM DATA SUMMARY

| Run/Train | Sampling Time θ (min) | Gas Volume $V_m$ (ft$^3$) | Liquid Volume $V_{lc}$ (ml) | Stack Temp. $T_s$ (°F) | Dry Gas Meter Temp. $T_m$ (°F) | Nozzle Diameter $D_n$ (in.) | Gas Velocity $V_s$ (ft/sec) | Moisture Content (%) | Pressure Drop ΔH (in. $H_2O$) | Percent Isokinetic Average |
|---|---|---|---|---|---|---|---|---|---|---|
| Run I | | | | | | | | | | |
| Stack | — | — | — | — | — | — | — | — | — | — |
| Combustion Zone | 120 | 116.4 | 103 | 1164 | 84 | * | — | 7.33 | 3.5 | — |
| Run II | | | | | | | | | | |
| Stack | 60 | 54.5 | 552 | 130 | 75 | 3/8 | 27.74 | 30.6 | 3.5 | 106 |
| Combustion Zone | 120 | 132.1 | 107 | 1089 | 93 | * | 80 | 6.46 | 3.5 | — |
| Run III | | | | | | | | | | |
| Stack | 60 | 36.5 | 205 | 135 | 88 | 3/8 | 32.25 | 33.7 | 1.5 | 99 |
| Combustion Zone | 113 | 129.6 | 246 | 1046 | 91 | * | 90 | 5.48 | 2.2 | — |

*No nozzle was used for the combustion zone sampling train.

TABLE B-2. ROLLINS SAMPLE GAS VOLUMES AT STANDARD CONDITIONS

| Test No. | Stack |  |  |  | Hot Zone |  |  |  |
|---|---|---|---|---|---|---|---|---|
|  | Dry |  | Wet |  | Dry |  | Wet |  |
|  | $ft^3$ | $m^3$ | $ft^3$ | $m^3$ | $ft^3$ | $m^3$ | $ft^3$ | $m^3$ |
| I | - | - | - | - | 113.70 | 3.22 | 132.43 | 3.65 |
| II | 54.82 | 1.55 | 78.99 | 2.24 | 128.06 | 3.63 | 136.88 | 3.88 |
| III | 36.80 | 1.04 | 54.97 | 1.56 | 125.29 | 3.55 | 136.90 | 3.88 |

TABLE 6-3. COLLECTED WATER VOLUME DATA

| Test | Water Volumes in Impingers |  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|
|  | 1st Imp. (ml) |  | 2nd Imp. (ml) |  | 3rd Imp. (ml) |  | 4th Imp. (g) |  | Total Collected Liquid Sample (ml) |
|  | Initial | Final | Initial | Final | Initial | Final | Initial | Final |  |
| I Hot Zone | 100 | 270 | 100 | 90 | – | 5 | – | 30 | 185 |
| Stack | – | – | – | – | – | – | – | – | – |
| II Hot Zone | 100 | 190 | 100 | 145 | – | 10 | – | 42 | 167 |
| Stack | 100 | 405 | 100 | 270 | – | 15 | – | 22 | 512 |
| III Hot Zone | 100 | 245 | 100 | 140 | – | 10 | – | 51 | 246 |
| Stack | 100 | 410 | 100 | 160 | – | 5 | – | 10 | 385 |

# APPENDIX C

## ANALYTICAL CHEMISTRY DETAILS - PCB TESTS

Many of the analyses of the samples from the PCB tests were performed by subcontracted laboratories. All sample preparation as described in Section 4.1.4.1 was carried out in the TRW chemistry laboratories. In addition, the gravimetric and IR organic survey techniques were performed at TRW. The following laboratories were used for the analyses indicated:

- Ultrachem - quantitative analysis for PCBs by GC/MS.
- West Coast Technical Service, Inc. - qualitative organic surveys by LRMS.
- Barringer Research Ltd. - quantitative inorganic surveys by ICPOES.
- Commercial Testing and Engineering - semi-quantitative inorganic surveys by SSMS.

Ultrachem, located in Walnut Creek, California, was utilized for its experience and demonstrated ability in PCB analyses. They used a Finnigan Model 9500 gas chromatograph connected, via a single stage glass jet separator, to a Finnigan Model 3100D quadrupole mass spectrometer controlled by a System Industries System/250 computer system.

Five µl of each sample was injected onto a 5 ft x 1/4" OD OV-17 on 100/120 mesh Chromosorb W AW-DMCS column, temperature programmed from 125°C to 250°C at 8°C/min. The vacuum divertor valve (solvent dump valve) was on for 1-1/2 minutes after injection; an additional 30 seconds was allowed for the column flow to equilibrate before temperature programming and mass spec scanning were started. The following respective sets of ions were used for the detection of the monochloro through octachlorobiphenyls - 188-198, 222-232, 256-266, 290-300, 324-334, 358-368, 392-436. Integration time was 17 milliseconds per atomic mass unit; the seven sets of ions were scanned every 1.9 seconds in a cyclic, continuous recording. The procedure yielded a detection limit of 3 to 5 ng for a 5 µl injection. Every sample derived from the PCB tests at Rollins was analyzed by Ultrachem, including all blanks, controls, and standards.

West Coast Technical Service (WCTS) in Cerritos, California, is frequently used for LRMS and other analytical work. In this particular case, TRW's mass spectrometer was down because of facility modifications underway to create a mass spectrometry center to house the Hitachi Perkin-Elmer RMU-6 instrument and TRW's new Finnigan GC/MS system. Organic survey samples were thus sent to WCTS, who also has a Hitachi Perkin-Elmer RMU-6 MS instrument.

Barringer Research in Rexdale, Ontario, Canada, was utilized to perform inorganic analyses because their ICPOES technique provides data on a relatively large number of elements (32) with good accuracy and at very low cost. Analyses performed by Barringer on samples from waste test burns at Systems Technology and St. Lawrence Cement (facility nos. 3 and 7) yielded data that was on the average 14% lower than AAS data with a range from -56% to +68% using 30 data points. Because of the good accuracy obtained on these previous samples, which were in the same or similar solution matrices, and the generally low concentrations found, no AAS work was performed on the Rollins samples.

Commercial Testing and Engineering (CT&E) located in Golden, Colorado, was selected to run complete inorganic surveys by SSMS on samples of dissolved solid residues from the Rollins kiln. SSMS is a very specialized and expensive analysis technique that is available from only a few commercial laboratories in this country. CT&E is one of these labs and also has experience with analyzing environmental samples.

# APPENDIX D

## PCB ANALYSIS BY GC/MS

The analysis of the Rollins sample concentrates for PCBs was carried out using the method in this appendix as a guide. The instrument and data system used in the analysis was not the same as the system for which the method is written so slight changes in the method were necessary. In particular, an EPA Aroclor standard of known PCB composition was used for determination of response factors.

## D.1 SCOPE AND APPLICATION

### Scope

This method is a direct analysis for polychlorinated biphenyl compounds (PCB). A search for these compounds is carried out primarily because of 1) their carcinogenic properties and 2) their tendency to be bio-accumulative and to resist degradation. If these materials are found to exist in environmental samples at significant levels (to be defined later), it is almost certain that a Level 2 analysis will follow.

### Sensitivity

The actual instrument sensitivity is expected to vary with factors including operating parameters and the efficiency of the GC/MS interface/sample enrichment device. In practice 5-10 nanograms (ng) will yield an MS signal with a usable signal noise ratio of >10:1. A dynamic range of approximately 1000 is typical. If the detectors are saturated a sample dilution is required.

### Detection Limit

There will typically have to be at least 50 μg of a PCB compound extracted from a sample and concentrated to 10 ml final sample solution. This presumes a 1 μl sample injection volume and the typical instrument sensitivity specified above. Larger sample injection volumes are possible.

### Interferenc

The possibility of non-PCB compounds interfering with the analysis by causing a false positive reading is about 3 percent. This is based on the number of non-PCB compounds which have a spectrum containing one or more of the specific PCB subset ions in the 10-100 percent relative abundance range. No interferences have been identified which would mask the presence of PCB. False positives are easily detected by the additional printed out data during the quantification step.

## D.2 SUMMARY OF METHOD

This is a combined gas chromatography/mass spectrometry method (GC/MS). Microliter quantities of concentrated sample extracts are injected into the chromatograph. The concentrated extracts result from the extraction of the various samples obtained from the sampling activity.

The extraction and concentration procedures are specified in Sections 7.3 and 7.4 of this document.

Microliter sized samples are injected onto a gas chromatographic column and are separated by the differences in the retention characteristics between the sample components and the column material. As the mixture components elute from the column they are transported via an instrument interface to a mass spectrometer preprogrammed to act as a detector specifically for PCBs. The signal from the mass spectrometer is converted into a mass fragmentogram, a specialized gas chromatogram. The presence of peaks at levels significantly greater than those for the reagent blank samples indicate PCB presence at high confidence levels. These levels are quantitatively determined using external standardization.

## D.3 DEFINITIONS

- Subset masses - a group of ions whose masses are characteristic to a particular class of compounds.
- Mass fragmentogram - a gas chromatogram, using the MS as a detector for specifically selected fragment ions.
- TIM - Total Ion Monitoring
- SIM - Selective Ion Monitoring
- PCB - Class - Group of PCB having the same chlorine content.
- 1Cl-PCB, 2Cl-PCB, etc. - This refers to the number of chlorine atoms on the PCB molecule. The maximum number is ten.

## D.4 SAMPLE HANDLING AND PRESERVATION

These samples are organic solutions resulting from extraction and concentration. They are contained in stoppered or septum sealed ampules or flasks typically containing less than 5 ml. These samples should never be exposed to direct sunlight. These samples shall be stored in a refrigerator when not being used. Solvent losses through evaporation shall be minimized by maintaining good seals on the containers. Abnormal or unusual losses in solvent volume shall be reported on the data report sheet. Contact of the sample with hands and other sources of outside contamination shall be avoided. PCBs are toxic materials and due care shall be used in handling all samples and standards. Gloves and the fume hoods are appropriate considerations.

## D.5 APPARATUS

This section specifies the major pieces of required apparatus. A normal compliment of glassware and various laboratory implements is assumed.

Automated Combined GC/MS, Finnigan Model 40[illegible]

Several quadrapole or magnetic sector instruments, computer driven, are available to perform this analysis using the selective ion monitoring or SIM technique. Basic required capabilities include

- Resolution sufficient to obtain unit mass resolution in the 40-400 range. This is typically 1000.
- Capability for glass 2-4mm ID packed columns and a sample enrichment device to achieve the instrument sensitivities described above.
- Electron multiplier detection system.
- Selective ion monitoring capability for at least 3 selected ions.

Interactive Data System

Capable of gathering and storing mass abundance data from the MS, generating fragmentograms, and performing quantitation with peak area or peak height ratios, normalizing and correcting for background.

GC column, a 6-foot glass column containing 3 percent OV-17 on 100-200 mesh Gas-Chrom Q or equivalent.

GC injection syringes, 10 µl, Hamilton 701-N or equivalent.

Analytical balance, capable of weighing 0.05 mg.

Volumetric Flask, 2 ml, hexagonal base, Corning 5630 or equivalent.

## D.6 REAGENTS

Supplies of the following solvents, Pesticide Grade, Distilled-in-Glass ®, Nanograde ® or equivalent should be kept at hand in several liter quantities. These solvents will be used to prepare analytical standards, make dilutions, do solvent replacement or other similar activities as required.

- Pentane
- Methylene chloride
- Methanol
- Acetone
- Petroleum ether

- Hexane
- Di-ethyl ether

PCB isomers, 99+ percent purity. Available from Analabs, Inc., New Haven, Connecticut. The stock of available standard PCB isomers should include but not be limited to the following. There is no evidence of significant variation in instrument response with position isomers of PCBs with each chlorine level. Those on the list were selected on the basis of cost and availability. These compounds are used to determine response factors (instrument sensitivity) in the calibration steps.

3 - chlorobiphenyl

2, 3 - dichlorobiphenyl

2, 5, 4' - trichlorobiphenyl

2, 4, 2', 4' - tetrachlorobiphenyl

2, 4, 5, 2', 5' - pentachlorobiphenyl

2, 4, 6, 2', 4', 6' - hexachlorobiphenyl

2, 3, 4, 5, 6, 2', 5' - heptachlorobiphenyl

## D.7 PROCEDURE

### D.7.1 Preparation of the Unknown Samples

The samples in solution shall be prepared in such a way that the solutions will be concentrated as much as is practically possible: Take a 2 ml aliquot of the 10 ml sample resulting from the concentration of the sample extracts. Place it in a clean 2 ml volumetric flask or vial. This sample will be used for the µl injections. If more concentration is required, put the 2 ml in a receiver ampule and direct a stream of clean filtered laboratory air over the solution until the sample is at the desired volume.

### D.7.2 Preparation of PCB Standard Samples

Concentrated stock solutions of the desired individual PCB isomers shall be made. Five milligrams in 50 milliliters of iso-octane yields a suitable concentration of 100 nanograms (ng) per microliter (µℓ). Portions of these stock solutions can be serially diluted or combined with each other to produce working standard mixtures. The working standard solutions will be made up in 100 ml volumetric flasks. The attached table (courtesy of the EPA) provides a guide of volumes of stock solution required to yield 100 ml of the desired working standard solution.

### 0.7.3 Preparation of the GC/MS instrument

The chromatograph shall be prepared according to the manufacturer's operating manual. Key GC operating parameters to be used are as follows:

- Temperatures

  Oven - Programmed from 100°C to 275°C at 12°C/min.

  Injector - 200° to 220°C

  Transfer lines - 150° to 175°C

  Separator - 200° to 220°C

- Carrier gas - Helium at 30 ml/min.

- Solvent divert time programmed to divert solvent and other species until a preprogrammed start time.

- Other parameters may be added (to be determined).

The mass spectrometer (MS) shall be prepared according to the manufacturer's operating manual. Briefly summarized, this procedure includes:

- Monitoring all applicable pressures and confirming that proper vacuum levels are being achieved. Proper levels are specified in the manual.

- Checking all applicable temperatures and making adjustments as necessary:

  Ionizer - ~40°C

  Manifold - ~40°C

  Electron multiplier - ~40°C

- Checking performance of ionizer

  Emission current - 0.2 to 0.5 milliamps

- Optimizing resolution/sensitivity

  Electron multiplier voltage - 1500 to 2000 volts

- Confirming that all electronics associated with data gathering are functioning properly.

Date and record parameters in instrument logbook.

## Computer Data Gathering/Data Storage

The data acquisition and storage operations shall be preprogrammed to select only ions of specific mass to charge ratios that are characteristic of PCBs. Program the following data gathering parameters into the instrument controller.

a) Selected mass set voltages 188-198, 222-232, 256-266, 290-300, 324-334, 358-368, 392-436

b) Integration time at each mass set voltage = 17 msec

c) Samples per AMU = 1

d) Counts threshold = 200 to 300

e) Run time = 99 min (operator can intervene)

Date and record in instrument logbook.

### D.7.4 Starting the Analysis

When all preparations have been completed, inject the sample into the gas chromatograph which is set in the solvent divert mode. At a given time after injection, the solvent divert valve directs the sample components eluting from the GC into the MS. Concurrently, a signal is sent to start the MS scanning sequence and data acquisition of the mass spec/computer to acquire and store data in accordance with the commands inputed by the operator. The instrument will then gather and store data over the time interval during which the PCBs are expected to elute. The analysis will continue for a pre-set duration or until the operator intervenes. The system shall be programmed to stop the analysis after 99 minutes have elapsed from time of injection.

### D.7.5 Data Processing and PCB Quantification Calibration

A known mixture of the pure isomer standards (D.6) is injected into the chromatograph and analyzed using the same technique and operating parameters as used for the samples and which are described above. The instrument can detect as low as 3-5 ng but for calibration purposes, accurately injected amounts in the 100 ng range yield more accurate PCB response factors.

The reconstructed chromatogram based on the normalized raw data is obtained via computer interaction. The scan number or spectrum number corresponding to the top of each peak is noted. The spectrum number representing the top of a peak is inputed to the computer, and the command is given to obtain the raw portioned data counts for the selected AMUs corresponding to the molecular ion cluster for that PCB. These counts represent the number of ions actually detected for each AMU. Knowledge of the standard composition and retention times enables the operator to identify the level of chlorination represented in each peak, i.e., 1 Cl, 2 Cl, etc. The selected AMUs to be searched are relatively specific to each chlorine level, and these are tabulated below. For example, if the peak in the chromatogram of the standard is a 3 chlorine PCB, the 256-266 AMU range is selected but only the 256, 258, 260 AMU detected ions are summed.

The sum of the ions for the selected AMUs representing each PCB chlorine level is divided by the amount (ng) of each PCB standard injected into the instrument. A response factor of ion counts per nanogram is obtained for each of the PCB standards with a different chlorine level. This standard should be analyzed at least three times, average response factors determined and the standard deviation calculated. Thereafter, the standard is injected at the start of each working day to confirm acceptable instrument performance.

## D.8 QUANTIFICATION OF PCBs IN SAMPLES

At the completion of the analysis of a sample (mass spectral data in storage) perform the following:

1) Obtain the reconstructed chromatogram using all of the subset data specified in D.7.3. This signal normalized output provides retention time data from which the composition of the PCB (chlorine levels) can be estimated. This provides the data analyst a feel for the type of PCB mixture with which he is working (e.g., Aroclor 1242, 1260, etc.) but is not needed for final identification and quantification.

TABLE D-1.

AMUs to Be Searched for Raw Counts

| Chlorine Atoms per PCB Molecule | Selected AMUs |
|---|---|
| 1Cl | 190 |
| 2 | 222, 224, 226 |
| 3 | 256, 258, 260 |
| 4 | 290, 292, 294, 296 |
| 5 | 324, 326, 328, 330 |
| 6 | 358, 360, 362 |
| 7 | 392, 394, 396 |
| 8 | 426, 428, 430 |
| 9 | 460, 462, 464 |

2) Obtain separate computer prepared chromatograms for each PCB class, i.e., 1Cl-PCB, 2Cl-PCB, etc., up to the maximum number of chlorines indicated by the chromatogram obtained in Step 1 of this section. This is obtained by inputing only the AMUs specific to each PCB class from Table D-1 and obtaining a chromatogram from those AMUs only.

3) Starting with the chromatogram for detected PCBs with the smallest number of chlorines, locate by spectrum number the top of each peak. (For purposes of illustration, assume that 1 chlorine and 2 chlorine PCBs were not detected and hence one starts with 3 chlorine PCBs). For each spectrum number corresponding to the top of a peak, a numerical printout is obtained presenting the absolute value, i.e., number of ion counts for AMUs 256 through 266 (from Section D.7.3). The number of ion counts only for AMUs 256, 258, and 260 are summed for each peak. These ion counts are divided by the calibration response factor for 3Cl-PCB to obtain the weight of 3Cl-PCB having caused that peak. This weight is the amount of 3Cl-PCB injected into the instrument which caused that peak.

$$\text{wt 3Cl-PCB (ng)} = \frac{\Sigma \text{ counts}}{\text{Response counts/ng}} \qquad \text{Equation 1}$$

The weights of 3Cl-PCB causing each of the peaks in the 3Cl-PCB chromatogram are summed and multiplied by the appropriate aliquot or dilution factors to obtain the total weight of 3Cl-PCB isomers in the starting 10 ml sample volume:

$$\text{wt 3Cl-PCB(ng)} = \Sigma\text{3Cl-PCB(ng)} \times \frac{1}{(A)\mu l} \times \frac{10^3 \mu l}{ml} \times Bml \qquad \text{Equation 2}$$

where:

wt 3Cl-PCB(ng) = amount of 3Cl-PCB in the entire sample

Σ3Cl-PCB(ng) = the sum of 3Cl-PCB in each of the peaks in the 3Cl-PCB chromatogram.

Aμl = size of the injection (microliters)

Bml = volume of total sample (milliliters)

The weights of 1Cl-PCB, 2Cl-PCB, 4Cl-PCB, 5Cl-PCB, etc., up through 10Cl-PCB, if detected, are calculated in similar fashion.

# APPENDIX E

## SAMPLE VOLUME DATA - NCB TESTS

## TABLE E-3

## STACK SAMPLING DATA

| Date | Run Number | Stack Temp °C | Stack Pressure mm of Hg | Oxygen % | Carbon Dioxide % | Excess Air % | Moisture Content % | Average Stack Gas Velocity m/sec | Average Sampling Velocity m/sec | Isokinetic Average | Total Volume Sampled at STP 21°C + 760 (Wet Basis) s.cu.m. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 12/14 | R1(B)-S | 70 | 763 | 12.8 | 6.2 | 150 | 25.5 | 7.983 | 8.230 | 103.1 | 1.385 |
| 12/15 | R2-S | 59 | 766 | 12.8 | 6.1 | 150 | 29.9 | 8.779 | 8.791 | 100.1 | 1.940 |
| 12/17 | R3-S | 58 | 767 | 13.1 | 5.7 | 160 | 29.9 | 8.560 | 8.465 | 98.9 | 1.874 |
| 12/18 | R4-S | 62 | 764 | 12.4 | 6.3 | 140 | 30.1 | 8.709 | 8.621 | 99.0 | 1.882 |

## TABLE E-2
## HOT ZONE SAMPLING DATA

| Date | Run Number | Moisture Content % (vol) | Wet Basis Total Volume Sampled at 20°C and 760 S.cu.m |
|---|---|---|---|
| 12/14 | R1B-HZ | 7.0 | 5.048 |
| 12/15 | R2-HZ | Lost | Sample |
| 12/17 | R3-HZ | 7.1 | 4.846 |
| 12/18 | R4-HZ | 6.4 | 4.778 |

# TABLE E-3

## ESTIMATED TOTAL GAS EFFLUENT FLOW RATIO

|  | HZ |  | S |  |
|---|---|---|---|---|
| Run No. | SCF/Min | SCM/Min | SCF/Min | SCM/Min |
| R1 | 34066 | 964 | 44821 | 1269 |
| R3 | 37581 | 1064 | 50090 | 1418 |
| R4 | 37592 | 1064 | 50267 | 1423 |

# APPENDIX F

## ANALYTICAL CHEMISTRY DETAILS - NCB TESTS

F.1. Sample Codes, Preparation, and Analysis Procedures

F.2. Recovery Efficiencies for NCB

F.3. Gravimetric and Volumetric Data

F.4. Analytical Results for Representative Waste Feed

F.5. Analytical Results for Probe Washes and Filters

F.6. Analytical Results for Dry Impingers

F.7 Analytical Results for Sorbent Traps

F.8. Analytical Results for Impingers

F.9. Analytical Results for Scrubber Water

F.10. Analytical Results for Gas Bulb Samples

## F.1 SAMPLE CODES, PREPARATION AND ANALYSIS PROCEDURES

### F.1.1. Sample Codes

Samples are identified by multi-syllable codes in which:

- The first syllable identifies the test run:

  R1(B) = background test on 12-14-76
  R2 = unsuccessful HCB test on 12-15-76
  R3 = HCB test on 12-17-76
  R4 = HCB test on 12-18-76
  R0 = general sample such as blended representative waste feed

- The second syllable identifies the sampling location:

  -HZ- = hot zone (combustion zone)
  -S- = stack

- The third syllable identifies the type of sample or sample train component:

  -PW = probe wash
  -F = filter
  -I1 = dry impinger (See page )
  ST = sorbent trap
  I = impinger
  SI = fresh scrubber water
  SO = spent scrubber water
  GB = gas bulb

- A final syllable indicates the solvent used, if the sample is an organic extract.

### F.1.2. Sample Preparation Procedures

#### F.1.2.1. Probe Washes

The probe washes were transferred to tared glass evaporating dishes and and the solvent evaporated on a hot plate. The dishes and contents were then dried to constant weight in a desiccator over Drierite®*.

The residue in the evaporating dish, for the hot zone (HZ) probe washes only, was taken up in methylene chloride. Several small portions of solvent were used, with agitation in an ultrasonic bath to facilitate uptake of soluble portions of the residue. Each resulting methylene chloride suspension was combined with the corresponding hot zone filter for Soxhlet extraction as described below.

---

* Trademark of W.A. Hammond Drierite Company, Xenia, Ohio.

### F.1.2.2. Filters

The filters were dried to constant weight in a desiccator over Drierite®. The dried filters were photographed.

The filters for the hot zone only were folded and placed in preextracted (24 hours with methylene chloride) cellulose Soxhlet thimbles. The methylene chloride suspension of the corresponding probe wash sample (above) was poured through the thimble; the solvent which drained through was added to the boiling flask of each Soxhlet apparatus. The boiling flask was charged with 200 ml of methylene chloride. The extraction was allowed to proceed for 24 hours.

An empty, preertracted thimble was extracted as a blank.

### F.1.2.3. Dry Impinger

The "Dry Impinger" was an empty, standard-sized impinger, used between the filter and the sorbent trap in the hot zone sampling train at Rollins. The sample obtained from this train component consisted of a measured volume of condensate, combined with water, acetone, and pentane rinses of the impinger and connecting glassware.

In the laboratory, the total sample volume was measured. An aliquot of the sample was set aside for chloride and nitrate analysis. The remainder of the sample was transferred to a tared glass evaporating dish. The liquid was evaporated on a hot plate. The dish and contents were dried to constant weight in a desiccator over Drierite®.

The residue in the evaporating dish was taken up in methylene chloride. Several small portions of solvent were used, with agitation in an ultrasonic bath to facilitate uptake of soluble portions of the residue. The methylene chloride solutions were transferred to 10 ml volumetric flasks and made to volume with fresh solvent.

A blank consisting of 1:1:1 water:acetone:pentane was carried through the evaporation-solution procedure.

### F.1.2.4. Sorbent Traps

The sorbent traps were placed in the specially designed extraction apparatus shown in Figure F-1. Each trap was extracted for 24 hours with pentane and then for 24 hours with methanol. The extracts were not combined for analysis. An unused sorbent trap was extracted; this served as a blank.

### F.1.2.5. Impingers

Total volumes of the combined impinger solutions* and distilled water

---

* Not including the "dry" impinger of the hot zone sampling train. See F.1.2.3.

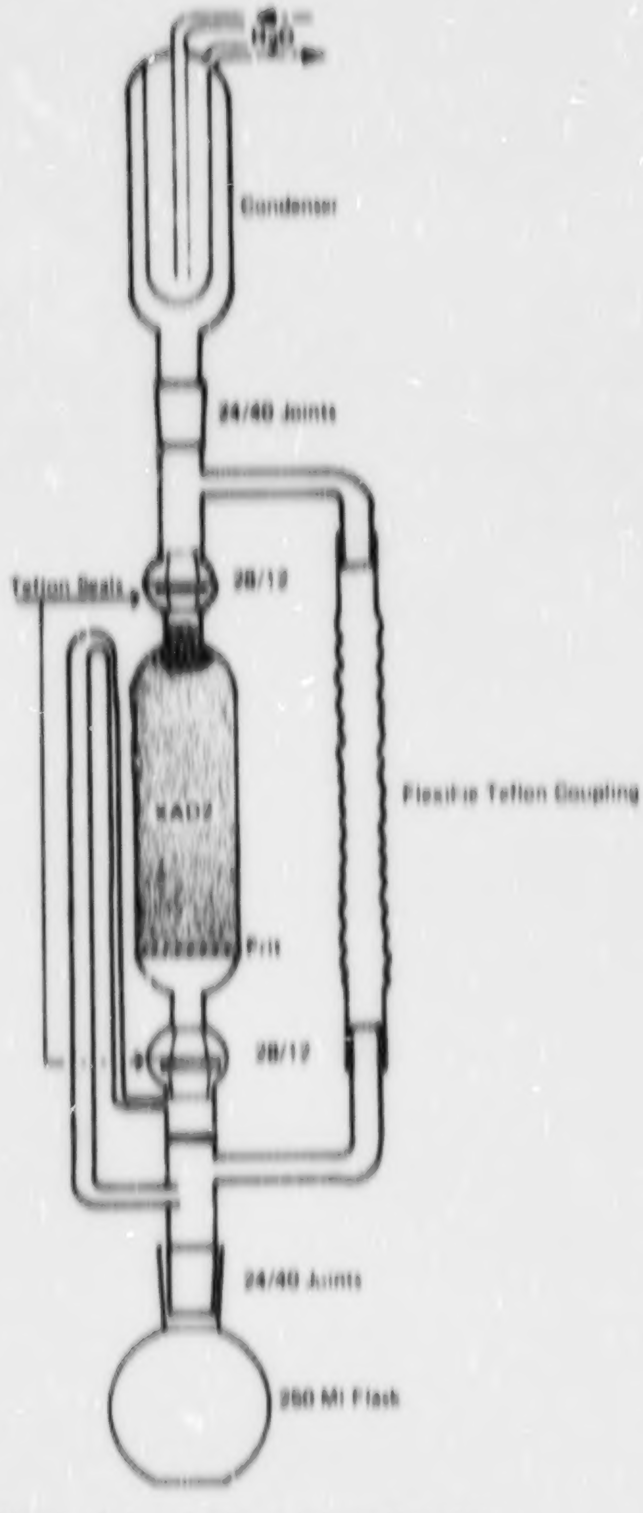

$H_2O$
Condenser
24/40 Joints
Teflon Seals
28/12
XAD2
Flexible Teflon Coupling
Frit
28/12
24/40 Joints
250 Ml Flask

Figure F-1. Sorbent trap extractor.

rinses of glassware from each sampling train were measured.

A portion of each solution was acidified to pH <2 (pH paper) with concentrated nitric acid. This acidified portion was refrigerated and stored in a Nalgene®* container.

A separate portion of each of the combined solutions was taken for chloride and nitrate analyses.

### F.1.2.6. Scrubber Water

The fresh scrubber water samples collected on 4 different test days were combined to give a single sample - RO-SI. A portion of this solution was acidified to pH <2 (pH paper) with concentrated nitric acid and refrigerated. A separate portion of the combined sample was taken for chloride and nitrate analyses. The pH of the combined sample was found to be 6.

The three spent scrubber water samples - R1(B)-SO, R3-SO, and R4-SO - were not combined. An acidified (pH <2) portion of each was stored in the refrigerator. A separate portion was taken for chloride and nitrate analyses. The pH's were measured and found to be 4 for R1(B)-SO; 3 for R3-SO; and 3 for R4-SO.

For determination of organics, 2 500-ml portions of each of the four scrubber water samples above and of a distilled water blank were extracted with 3 x 20 ml each of methylene chloride. The organic extracts were dried by passing them through anhydrous sodium sulfate.

## F.1.3. Analysis Procedures

### F.1.3.1. Gas Chromatographic Analysis of Unconcentrated Organic Extracts

The organic solvent extracts prepared, as described above, from the probe wash plus filter, dry impinger, sorbent trap and scrubber water from each test's sampling effort were analyzed by gas chromatography prior to any concentration step. The total volume of each extract was measured, and 5 µl portions were taken for injection.

The gas chromatographic conditions were as follows:

instrument = Varian 2700
detector = FID at 295°C
injector = glass lined, 275°C
column = 6' x 1/8" stainless steel packed with 10% OV-101 on 100/120 mesh Supelcoport®**

---

* Trademark of Nalge Company, Rochester, New York.

** Trademark of Supelco, Inc., Bellefonte, Pa.

program = Isothermal at 50°C for 3 min. Linear program
at 20°C/min to 275°C. Hold for 5-10 min at
275°C.

Quantitative calibration of the detector response was achieved by use of standard solutions of o- and p-nitrochlorobenzene. These two isomers were not resolved by the chromatographic conditions used. The retention time, relative to the leading edge of the solvent front, was found to be constant at 520 ± 12 sec. over the course of the analyses. The quantitative calibration changed somewhat from one week to the next. Typical calibration curves are shown in Figure F-2. Injections of calibration standards were interspersed with injections of samples each day.

### F.1.3.2. Concentration of Organic Extracts

After gas chromatographic analysis, each of the organic extracts was concentrated to a volume of <10 ml.* Concentration was accomplished by allowing solvent to evaporate from an open container under a gentle stream of nitrogen. The concentrated extracts were transferred to 10 ml volumetric flasks and the volume restored to 10 ml using fresh solvent.

### F.1.3.3. Gravimetric Quantification of Organic Extracts

A 5 ml aliquot (one half of total sample) was withdrawn by volumetric pipette from each 10 ml extract and transferred to a tared aluminum weighing dish. The contents of the dishes were allowed to evaporate in a hood at ambient temperature until constant weight (± 1 mg) was obtained (repetitive weighings at least 6 hours apart).

### F.1.3.4. Infrared Spectroscopy

A portion of each concentrated organic extract was taken for IR analysis. Spectra were obtained using KBr micropellets, or macropellets for the more concentrated solutions, using a Perkin-Elmer Model 521 grating spectrophotometer.

### F.1.3.5. Low Resolution Mass Spectrometry (LRMS)

A portion of each concentrated organic extract was taken for LRMS analysis. Analyses were done using both the batch inlet and the direct insertion probe of a DuPont (CEC) 21-110B high resolution mass spectrometer.

Both qualitative and quantitative results of the LRMS analyses are reported. The quantitative data given are expressed as estimated percent

---

* The I-1/$CH_2Cl_2$ extracts were initially 10 ml in total volume and were not concentrated further.

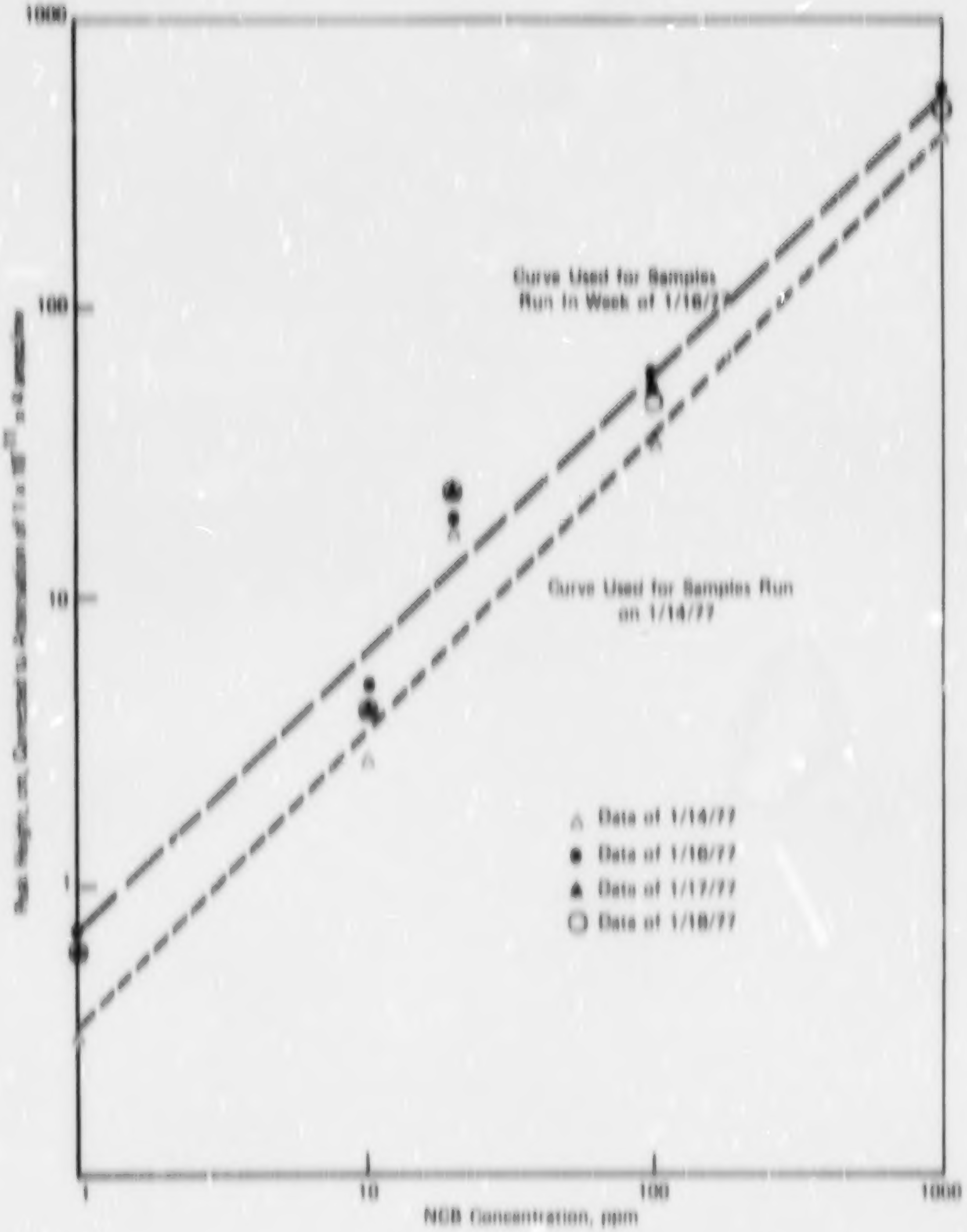


Figure F-2. GC calibration curve for NCB standards.

abundance of the indicated species, relative to the total quantity of sample which vaporized in the mass spectrometer inlet. They are not normalized to correct for nonvolatile portions of the sample. The precision of the estimated values varies with concentration; data for major components are probably reliable to within 50%. For components at very low concentration, the error may be as large as a factor of five.

Mass spectrometry on gas bulb samples was done by Gollub Analytical Services Corporation, Berkeley Heights, New Jersey.

### F.1.3.6. Elemental Analyses

These analyses were performed only on the waste feed and not on the effluent samples. Elemental analysis for probable major components (C, H, N, Cl, S) was done by Galbraith Laboratories, Knoxville, Tennessee.

Elemental analysis for trace components, especially metals, was done using spark source mass spectroscopy (SSMS) by Commercial Testing and Engineering Laboratories, Golden, Colorado. The sample was thermally low temperature ashed at 350°C for 1 hour in a laboratory furnace in a quartz crucible prior to analysis.

The ash content of the waste was determined at ADL by ashing in a muffle furnace at 850°C.

### F.1.3.7. Anion Analyses of Aqueous Samples

The stack and hot zone impinger solutions and the scrubber water samples were analyzed for chloride and nitrate.

For chloride analyses, a suitable aliquot was taken, oxidized with hydrogen peroxide, and treated with barium nitrate to precipitate sulfur species as barium sulfate. The resulting solution was then titrated using standardized mercuric nitrate titrant with S-diphenyl carbazone as the endpoint indicator. Standard 0.1 N hydrochloric acid was used as a reference standard.

For nitrate analyses, chloride was first removed by precipitation with silver sulfate. The samples were diluted to the proper range (0.1 to 2.0 ppm $NO_3^-$) and analyzed by the phenol disulfonic acid colorimetric method. The analysis was done on the Coleman Model 55 spectrophotometer at a wavelength of 410 nm. Reagent grade anhydrous sodium nitrate was used as a reference standard.

## F.2. RECOVERY EFFICIENCIES FOR NCB

To determine whether NCB could, in fact, be recovered if present in effluent samples, small quantities of NCB waste (taken from the survey sample) were spiked onto a sorbent trap and into some distilled water (simulated scrubber water). These samples were extracted as described above for the effluent samples and the extracts analyzed for NCB by quantitative gas chromatography.

For the sorbent trap, 90% recovery of NCB (8.97 mg out of a 10 mg spike) was found in the pentane extract. The subsequent methanol extract was found to contain no NCB (<0.2 mg or <2% of the spike).

For the aqueous solution, recoveries in the methylene chloride extracts were found to be: 86% at 1000 ppm NCB; 73% at 500 ppm NCB; and 66% at 100 ppm NCB.

## F.3. GRAVIMETRIC AND VOLUMETRIC DATA

Table F-1 presents the measured volumes for the various impinger solutions. These data are used in calculating the percent moisture in the sampled streams. The total final volume of solution is also used to calculate a mass emission concentration from the measured chloride concentration.

Table F-2 presents gravimetric data for the three types of sample (PW, F, and HZ-11) that were dried to constant weight before extraction. For each test and sampling location, the PW and F numbers in this table are the values which are summed to estimate the total particulate loading.

Table F-3 presents the results of gravimetric analyses on the various concentrated extracts. Also included are data for the various blanks which were run in parallel with the samples.

## F.4. ANALYTICAL RESULTS FOR REPRESENTATIVE WASTE FEED

It is necessary to distinguish between two different waste feed samples. The first, RO-REP(1), is the nitrochlorobenzene waste which was shipped by the waste producer. The second, RO-REP(2), is the blend of 20% (v) NCB:80% (v) #2 diesel oil which was actually fed to the incinerator in tests R3 and R4. A sample of unadulterated #2 diesel oil was also available; this is RO-REP(3).

All of the data imply that the "pure" NCB waste, as received in a heated tank truck, consisted mainly of nitrochlorobenzene. Analysis by GC, GC/MS and LRMS indicate that about 95% by weight of RO-REP(1) is nitrochlorobenzene. The IR spectrum is consistent with this, suggesting a mixture of nitrochlorobenzene isomers. Further confirmation is provided by the elemental analysis:

|  | C | H | N | Cl | S |
|---|---|---|---|---|---|
| Found for RO-REP(1) | 46.14 | 2.80 | 8.82 | 23.28 | 0.02 |
| Calc'd for $C_6H_4NO_2Cl$ | 45.66 | 2.56 | 8.92 | 22.3 | -- |

## TABLE F-1
## VOLUMES OF IMPINGER SOLUTIONS

| Run | Sample | Volume Charged Before Test ml | Volume Recovered After Test ml | Final Total Volume Including Rinses ml |
|---|---|---|---|---|
| R1(B) | HZ-II | 0 | 1[illegible]4 | 460 |
| | HZ-I | 2000 | 2075 | 2650 |
| | S-I | 250 | 550 | 645 |
| R3 | HZ-II | 0 | 164 | 370 |
| | HZ-I | 2000 | 2065 | 2540 |
| | S-I | 250 | 652 | 700 |
| R4 | HZ-II | 0 | 144 | 500 |
| | HZ-II | 2000 | 2055 | 2740 |
| | S-I | 250 | 663 | 700 |

## TABLE F-2
## GRAVIMETRIC DATA FOR PROBE WASH, FILTER, AND DRY IMPINGER SAMPLES

| Run | Sample | Residue After Drying to Constant Weight, mg. |
|---|---|---|
| R1(B) | HZ-PW | 83.9 |
| | HZ-F | 7.5 |
| | HZ-I1 | 164.6 |
| | S-PW | 47.3 |
| | S-F | 5.9 |
| R3 | HZ-PW | 334.0 |
| | HZ-F | 130.5 |
| | HZ-I1 | 400.9 |
| | S-PW | 5.5 |
| | S-F | 20.5 |
| R4 | HZ-PW | 240.0 |
| | HZ-F | 132.8 |
| | HZ-I1 | 364.9 |
| | S-PW | 11.6 |
| | S-F | 19.3 |

## TABLE F-3
## RESULTS OF GRAVIMETRIC ANALYSES ON CONCENTRATED ORGANIC EXTRACTS

| Run | Sample | Total Weight* mg |
|---|---|---|
| R1(B) | HZ-PWF/$CH_2Cl_2$ | 48.4 |
| | HZ-II/$CH_2Cl_2$ | 6.8 |
| | HZ-ST/Pentane | 23.8 |
| | HZ-ST/Methanol | 97.4 |
| | SO/$CH_2Cl_2$ | 0.4 |
| R3 | HZ-PWF/$CH_2Cl_2$ | 76.2 |
| | HZ-II/$CH_2Cl_2$ | 1.6 |
| | HZ-ST/Pentane | 3.4 |
| | HZ-ST/Methanol | 150.0 |
| | SO/$CH_2Cl_2$ | 0.0 |
| R4 | HZ-PWF/$CH_2Cl_2$ | 53.2 |
| | HZ-II/$CH_2Cl_2$ | 47.2 |
| | HZ-ST/Pentane | 9.0 |
| | HZ-ST/Methanol | 53.8 |
| | SO/$CH_2Cl_2$ | 0.4 |
| R0 | PWF Thimble Blank/$CH_2Cl_2$ | 1.2 |
| | II Mixed Solvent Blank/$CH_2Cl_2$ | 5.8 |
| | ST Blank/Pentane | 1.2 |
| | ST Blank/Methanol | 2.0 |
| | SI/$CH_2Cl_2$ | 2.0 |

* Based on value obtained by evaporating one half of total extract to dryness.

The density of the waste was measured and found to be 1.329 g/ml. Comparison with literature values* (1.368 for ortho, 1.534 for meta and 1.520 for para-nitrochlorobenzene at 20 ± 2°C) suggests that the ortho isomer is predominant.

Other species which were identified in the RO-REP(1) sample of undiluted NCB waste were dinitrochlorobenzene (about 3%) and nitrobenzene (about 1%). The LRMS also indicated a homologous series of compounds (about 3% total) which appear to be styrene-substituted di-tolyl ethers. The lowest member of this series has MW 198 and a molecular formula, confirmed by HRMS peak matching, of $C_{14}H_{14}O$. Others are at 302, 406, and 510 MW. Very small amounts of hexachlorobutadiene and hexachlorobenzene were also detected.

The waste had a low ash content; the loss on ignition was 99.2%

The only elements which were found by SSMS to be present in the waste at concentrations (μg/ml) greater than 1 ppm were: calcium, chlorine, iron and magnesium (all at ~1000), sodium (9), silicon (6), sulfur (6), phosphorus (5), potassium (4), copper (4), aluminum (3), titanium (2), nickel (1) and chromium (1). None of these are present at high enough concentration to cause concern for emissions of toxic metals at the actual feed rates used in the Rollins tests.**

The #2 diesel oil (RO-REP(3)) was not subjected to detailed analysis. A gas chromatographic analysis of a diluted sample showed a typical diesel oil profile. The density was measured and found to be 0.862 g/ml. An elemental analysis revealed the following composition: C, 84.31%; H, 12.57%; N, 0.2%; Cl, 0.71%; and S, 0.14%.

In most significant aspects, the blended waste feed sample, RO-REP(2), conformed to the properties expected for a 20% (v:v) mixture of the NCB waste, RO-REP(1), and #2 diesel oil, RO-REP(3). For example, the density of a 20% by volume blend was calculated to be 0.955 g/ml; the value found was 0.949. Quantitative analysis by gas chromatography indicated that the blend was 19% by volume NCB.

Because of the substantially different densities of the two components, a 20% by volume NCB mixture would be 27.8% by weight of NCB. The LRMS analysis of the RO-REP(2) sample led to an estimate of 28% NCB which again confirms the presumed composition of the blend. When the elemental analysis results were examined, however, an inconsistency appeared in the data.

---

* CRC Handbook of Chemistry and Physics, 43rd. ed., Chemical Rubber Publishing Co., Cleveland, Ohio, 1961, pp. 832-3.

** Given the actual feed rates and total gaseous emission rates, 1 μg/ml of any element in the NCB waste corresponds to a stack emission rate on the order of 0.004 mg/m³.

Samples of REP(1), REP(2) and REP(3) were resubmitted for analysis and the original results were confirmed. As indicated below, the blended waste sample has almost 50% more chlorine content than would be expected for a 27.8% by weight (20% by volume) mixturr of NCB with #2 diesel oil.

| | C | H | N | Cl | S |
|---|---|---|---|---|---|
| Calc'd. for 27.8% mix | 73.70 | 9.86 | ≤2.49 | 6.98 | 0.11 |
| Found for RO-REP(2) | 73.84<br>73.95 | 10.08<br>10.14 | 2.48<br>2.68 | 9.89<br>10.06 | 0.10<br>0.11 |
| Calc'd. for 41.4% mix | 66.34 | 8.53 | ≤3.70 | 10.05 | 0.09 |

Data for the other elements analyzed in the mixture are consistent with the 27.8% by weight composition of the blend. In order to account for the 10% chlorine found in the REP(2) sample, it would be necessary to postulate a mixture which was more than 40% by weight of NCB. The latter mixture would not have the carbon, hydrogen or nitrogen content consistent with that found in the blend.

The reason for the discrepancy between observed and expected chlorine content has not been identified. It does not arise from errors in the Galbraith analyses or in post-sampling contamination of the ADL samples, since Rollins' analysis at the time of testing showed 10.2% chlorine. It is improbable that the entire batch of blended waste could have been contaminated in a way which would explain the observations. In the absence of further information, the mean empirical value of 10.0% chlorine in the blend has been assumed to be the most reliable estimate of the true composition.

The LRMS analysis of the blended waste feed revealed, in addition to the 28% nitrochlorobenzene, 20% alkylnaphthalenes and 49% other hydrocarbons, mainly aliphatic, up to $C_{30}$, as major components. These are expected components of the diesel oil. The ether-styrene series of compounds was present at the expected 1% level. In addition, about 0.1% by weight of PCB's was detected in the mixture. The PCB's were sought and found at about the same level in the RO-REP(3), #2 diesel oil sample.

## F.5 ANALYTICAL RESULTS FOR PROBE WASHES AND FILTERS

### F.5.1 Quantitative Analysis by Gas Chromatography

Waste Components: No peaks were observed in the retention time range 520 ± 25 sec. relative to the solvent front (for NCB standards, R.T. = 520 ± 12 sec.). The quantity of NCB in these samples was therefore less than the limit of detection, or <0.2 mg in each sample.

Other Material: The detector response, after elution of solvent, was integrated for peaks with retention times (RT) up to 900 sec. Major peaks, equivalent quantities as NCB, and total chromatographic material as NCB were:

| R1(B) |  | R3 |  | R4 |  |
|---|---|---|---|---|---|
| RT | mg. as NCB | RT | mg. as NCB | RT | mg. as NCB |
| 750 | 2.5 | 755 | 2.6 |  |  |
| 781 | 1.4 | 784 | 1.6 | 773 | 2.4 |
| 791 | 1.3 |  |  |  |  |
| 815 | 5.4 | 820 | 5.1 | 810 | 4.1 |
| 868 | 1.3 | 863 | 3.6 | 818 | 1.2 |
| 890 | 2.4 | 895 | 3.1 | 843 | 4.0 |
| TOTAL | 19.4 | TOTAL | 17.8 | TOTAL | 13.0 |

## F.5.2 Qualitative and Quantitative Analysis by IR and LRMS

The IR of the Soxhlet thimble blank showed only weak aliphatic $CH_2$ and $CH_3$ bands. The LRMS of this sample showed:

| | |
|---|---|
| Fatty acids | ~26% |
| Homologous $C_nH_{2n}$ series | ~21% |
| Phthalates | ~20% |
| Other hydrocarbons | ~13% |
| MW 198, $C_{14}H_{14}O$ (ditolyl ether) | ~11% |
| Triglycerides | ~9% |

The IR of the R1(B)-H1-PWF/$CH_2Cl_2$ sample revealed strong bands characteristic of diesel fuel, moderate carbonyl bands at 1712 and 1740 cm$^{-1}$, and weak alkene bands. The LRMS of this sample showed:

| | |
|---|---|
| Homologous $C_nH_{2n}$ series, n even | ~ 38% |
| Homologous $C_nH_{2n}$ series, n odd | ~ 35% |
| $C_{16}$, $C_{18}$ Fatty Acids | ~ 25% |
| Phthalates | ~ 2% |

For the R3 test, the IR of the PWF/$CH_2Cl_2$ extract was similar to that of the corresponding background test, except that the carbonyl band was weaker and a weak band attributable to silicone grease was observed. Silicones (~4%) were confirmed by LRMS. The LRMS also showed:

|  |  |
|---|---|
| Homologous $C_nH_{2n}$ series, n even | ~65% |
| Paraffin, $C_{20}$-$C_{40}$ | ~27% |
| $C_{16}$, $C_{18}$ Fatty acids | ~6% |

The R4 PWF sample was like the R3 in the IR spectrum, but showed evidence of possible nitrogen groups ($-NO_2$, $-NH_2$, or nitrates) and aromatic ring stretch. The LRMS spectrum was dominated by one major component which appeared to be hydroxy octoxy benzophenone (MW 326):

|  |  |
|---|---|
| MW 326, $C_{21}H_{26}O_3$ | ~69% |
| MW 346, $C_{24}H_{42}O$ (346.3253) | ~13% |
| $C_{16}$-$C_{18}$ Fatty acids | ~6% |
| Nonyl Phenol | ~5% |
| m/e 530.47897 (?) | ~5% |
| Triglycerides | ~1% |

## F.6. ANALYTICAL RESULTS FOR DRY IMPINGERS

### F.6.1. Quantitative Analysis by Gas Chromatography

Waste components: In the retention time range 520 ± 25 sec, relative to the solvent front (for HCB standards, R.T. = 520 ± 12 sec), one peak was observed in the R1(B) sample chromatogram and one in the R4 sample. The areas, however, corresponded to concentrations of < 2.2 ppm of HCB or <0.02 mg in the total extract. No peaks in the 520 ± 25 sec. range were found for R3-HZ-I1/$CH_2Cl_2$.

Other Material: Total detector response for all three chromatograms was equivalent to <1.0 mg, quantified as HCB. The largest peak in the chromatogram in each of the I1/$CH_2Cl_2$ samples had a retention time of 390 ± 10 sec.

### F.6.2. Quantitative and Qualitative Analysis by IR and LRMS

The IR spectrum of the mixed-solvent blank suggested diesel fuel and aromatic compounds, possibly phenol derivatives. The LRMS indicated 56% of this sample was a species tentatively identified as hydroxy octoxy benzophenone (MW 326). Other LRMS indications were:

|  |  |
|---|---|
| MW 326 | ~56% |
| Triglycerides | ~14% |
| Other hydrocarbons | ~12% |
| Nonyl phenol | ~9% |
| m/e 530 | ~5% |
| $C_{16}$, $C_{18}$ Fatty acids | ~3% |
| Phthalates | ~1% |

For the background test, R1(B), the IR of the dry impinger sample showed sulfate and bisulfate (strong) and weak hydrocarbon and OH, NH bands. The LRMS of this sample showed almost nothing except silicones and sulfate decomposition products.

For the R3-HZ-II/$CH_2Cl_2$ sample, the IR showed only weak bands for aliphatics, -OH or -NH, and silicones. The LRMS revealed nothing, too little sample volatilized.

The corresponding R4 sample looked more like the mixed-solvent blank by both techniques. The IR's were very similar. The LRMS showed:

| | |
|---|---|
| Triglycerides | ∿44% |
| Silicones | ∿26% |
| Hydroxy Octoxy Benzophenone? (MW 326) | ∿22% |
| $C_{16}$,$C_{18}$ Fatty acids | ∿5% |
| Other Hydrocarbons | ∿3% |

### F.6.3. Analyses for Chloride and Nitrate

The results of chloride and nitrate analyses for the dry impinger samples were:

| Sample | $Cl^-$ (ppm) | $NO_3^-$ (ppm) |
|---|---|---|
| R1(B) - HZ-II | 4850. | 90. |
| R3-HZ-II | 64200. | 2010. |
| R4-HZ-II | 56800. | 4780. |

## F.7. ANALYTICAL RESULTS FOR SORBENT TRAPS

### F.7.1. Quantitative Analysis by Gas Chromatography

Waste components: In the pentane extracts of the sorbent traps, a few peaks were observed in the retention time range 520 ± 25 sec. relative to the solvent front (for NCB standards, R.T. = 520 ± 12 sec). Using the NCB quantitative calibration curve, the calculated quantities of material in this retention time range were:

| | |
|---|---|
| R1(B) | 0.06 mg |
| R3 | 0.02 mg |
| R4 | <0.01 mg |

In the methanol extracts of the sorbent traps there were no peaks in the 520 ± 25 sec. retention time range.

Other material: The detector response, after elution of solvent, was integrated for peaks with retention times up to 900 sec. Predominant peaks, equivalent quantities as NCB, and total chromatographable material as NCB were, for the pentane extracts:

| R1(B)-HZ-ST/Pentane | | R3-HZ-ST/Pentane | | R4-HZ-ST/Pentane | |
|---|---|---|---|---|---|
| RT, sec | mg as NCB | RT, sec | mg as NCB | RT, sec | mg as NCB |
| 390 | 3.8 | 370 | 0.4 | 380 | 0.4 |
| 495 | 2.6 | 460 | 0.2 | 411 | 6.9 |
| 580 | 1.3 | | | 492 | 2.8 |
| 650 | 0.7 | | | 579 | 0.8 |
| 710 | 0.3 | | | 684 | 0.4 |
| 780 | 0.2 | 825 | 2.4 | 765 | 0.5 |
| TOTAL | 14.4 | TOTAL | 3.8 | 778 | 0.5 |
| | | | | 830 | 3.6 |
| | | | | 886 | 5.2 |
| | | | | TOTAL | 23.5 |

For the methanol extracts, no peaks were observed in the sample chromatograms that were not present in the RO-ST/methanol blank. The total areas of peaks eluting after the solvent but in less than 900 sec. were (as NCB):

| | |
|---|---|
| R1(B)-HZ-ST/methanol | 1.8 mg |
| R3-HZ-ST/methanol | 1.2 mg |
| R4-HZ-ST/methanol | 0.4 mg |

### F.7.2. Quantitative and Qualitative Analysis by IR and LRMS

#### Pentane Extracts

The IR spectrum of the ST/Pentane blank showed moderate intensity bands characteristic of hydrocarbons, and weak bands characteristic of a carbonyl (1730 cm$^{-1}$), silicon grease, and -OH or -NH. The LRMS on this blank showed:

| | |
|---|---|
| Paraffins | ≈77% |
| Napthalene | ≈10% |
| Alkyl Napthalenes | ≈ 4% |
| Anthracene | ≈ 1% |
| MW 340 ($C_{22}H_{28}O_3$) | ≈ 5% |
| Triglycerides | ≈ 2% |
| m/e 530 | ≈ 1% |

For the R1(B) background test, the IR spectrum of the ST/Pentane extract was similar to that of the blank: silicone grease (moderate); hydrocarbon (moderate); -OH or -NH (weak) and carbonyl at 1740 cm$^{-1}$ (weak). The LRMS on this sample suggested the following:

| | |
|---|---|
| Silicones | ≈40% |
| Benzoic Acid | ≈23% |
| Aliphatic Hydrocarbons, m/e 400-470 | ≈15% |
| $C_{13}$, $C_{15}$, $C_{17}$ Fatty Acids | ≈10% |
| Dibutyl tin Chloride | ≈11% |

The presence of the organic tin compound was unexpected. Its identity was confirmed by HRMS peak matching as well as by the distinctive tin isotope pattern. It is expected that this compound may be due to carry-over or incomplete purging of the Rollins facility, since "metal alkyls" were among the wastes burned at the facility during the week of ADL's testing.

The IR spectrum of the R3-HZ-ST/Pentane extract was similar to that of the background sample: silicone grease (strong), hydrocarbon (moderate), -OH or -NH (moderate), carbonyl at 1730 cm$^{-1}$ (moderate) and alkene or C-Cl (weak). The LRMS showed mostly benzoic acid:

| | |
|---|---|
| Benzoic Acid | ≈85% |
| Alkyl Benzenes | ≈ 9% |
| Toluic Acid | ≈ 2% |
| Ethyl Benzoate | ≈ 2% |
| $C_{10}H_{13}O_2Cl$ | ≈ 1% |
| Phenol | ≈ 1% |
| Silicone | ≈ 1% |

For the R4 test, the IR spectrum was much like that of the R3 sample, except that the hydrocarbon bands were stronger and the carbonyl band appeared at 1700 cm$^{-1}$. The LRMS indicated that, as in the background test, dibutyl tin chloride was a significant component:

| | |
|---|---|
| Dibutyl Tin Chloride | ∿46% |
| Aliphatic Hydrocarbons | ∿24% |
| Silicone | ∿20% |
| $C_{12}$, $C_{14}$, Fatty Acids | ∿ 6% |
| Alkyl Napthalenes | ∿ 2% |
| Anthracene | ∿ 2% |

Again, it is suspected that the organic tin compound is probably a carry-over from a prior Rollins waste burn.

Methanol Extracts

The RO-ST/methanol blank had an IR spectrum with moderate intensity bands for aliphatic $CH_2$, $CH_3$, -OH, or -NH, $SO_4^=$ and $CO_3^=$, and weak bands for amides. The LRMS indicated:

| | |
|---|---|
| Benzoic Acid | ∿63% |
| MW 326 (hydroxy octoxy benzophenone) | ∿24% |
| Toluic Acid | ∿ 4% |
| $C_{16}$, $C_{18}$ Fatty Acids | ∿ 4 |
| Ethyl Benzoate | ∿ 4% |
| m/e 530 | ∿ 1% |

For the background test, the ST/methanol extract IR spectrum showed moderate intensity hydrocarbon and -OH or -NH bands and weak bands indicating an ether or alcohol. There was also strong evidence of an amide linkage. The LRMS of this sample showed very substantial amounts of HCl (∿78% of the total LRMS intensity). Other LRMS indications were:

| | |
|---|---|
| Sulfate Decomposition Products | ∿ 8% |
| Dihydrofuran, MW 70 | ∿ 4% |
| Furfural or Furan Aldehyde | ∿ 4% |
| m/e 84 (possible $C_5H_8O$) | ∿ 3% |
| Phthalates | ∿ 2% |
| Dibutyl Tin Chloride | ∿ 1% |

For the R3- and R4- HZ- ST/methanol, the IR spectra were the same as that of the corresponding R1(B) sample. The LRMS results for the R3 and R4 methanol extracts were:

| | R3 | R4 |
|---|---|---|
| HCl | ∿64% | ∿36% |
| Various N- Containing Decomposition Products ($C_{10}H_{14}N_2O_2$, $C_4H_8NO_2$, $C_4H_8N$) | ∿16% | ∿9% |
| Unidentified (numerous small peaks) | ∿16% | ∿48% |
| Sulfate Decomposition Products | ∿ 3% | ∿ 2% |
| Phthalates | ∿ 1% | |
| Dibutyl Tin Chloride | ∿ 1% | |
| Benzoic Acid | | ∿ 2% |
| Methyl Benzoate | | ∿ 4% |

The species referred to as "decomposition products" in the LRMS data summary are so called because they appear at much higher probe temperatures (∿300°C) than is consistent with their low molecular weight. These species are apparently formed by pyrolysis of the sample in the heated probe.

## F.8 ANALYTICAL RESULTS FOR IMPINGERS

The impinger solutions originally charged at the start of sample collection were 0.1N sodium acetate for all stack samples and the R1(B) hot zone sample and 5N sodium acetate for the R3 and R4 hot zone samples. The results of the chloride and nitrate analyses were:

| Sample | $Cl^-$ | $NO_3^-$ |
|---|---|---|
| R1(B) - S - 1 | 5.5 | 0.7 |
| R1(B) -HZ - 1 | 120.0 | 41.8 |
| R3 - S - 1 | 11.5 | 5.2 |
| R3 - HZ - 1 | 685.0 | 45.0 |
| R4 - S - 1 | 35.0 | 4.0 |
| R4 - HZ - 1 | 1775.0 | 82.0 |

## F. 9 ANALYTICAL RESULTS FOR SCRUBBER WATER

### F.9.1 Quantitative Analysis of Organic Extracts by Gas Chromatography

No peaks were found in any of the samples in the retention time range (820 ± 25 sec) which included the retention time of NCB. There were no significant peaks in the R3- or R4- SO/$CH_2Cl_2$ extracts that were not present in the S1 or blank extracts. The only significant peak in all of the chromatograms had a retention time of 700 sec relative to the solvent front and an intensity corresponding to <1 mg as NCB.

### F.9.2 Qualitative and Quantitative Analysis by IR and LRMS

The IR spectrum of the fresh scrubber water extract (RO-SI/$CH_2Cl_2$) showed bands characteristic of sulfate (very strong), $CH_2$, $CH_3$ (weak), -OH or -NH (weak) and carbonyl at 1730 $cm^{-1}$ (moderate). The LRMS of this extract showed:

| | |
|---|---|
| Nonylphenol Dimer | ~33% |
| Unidentified (numerous peaks) | ~25% |
| Nonylphenol | ~23% |
| Phthalate | ~10% |
| Piperidine | ~ 8% |
| Fatty Acids | ~ 1% |

The IR spectra of the R1(B)-, R3-, and R4- SO/$CH_2Cl_2$ extracts were very similar to those of the RO-SI extract, except that some silicone bands were observed. The LRMS spectra, again, were similar to those of the SI-extract. The LRMS spectra did indicate the presence of the ditolylether species, previously identified in the representative waste feed. Some mono- and di- brominated analogs appeared to be present in the R1(B)- SO/$CH_2Cl_2$ extract. Nitrogen-containing species amounted to about 15% of the total LRMS intensity for the spent scrubber water effluents.

### F.9.3 Analyses for Chloride and Nitrate

| Samples | $Cl^-$, ppm | $NO_3^-$, ppm |
|---|---|---|
| RO - SI | 23.5 | 0.3 |
| R1(B) - SO | 208 | 47.8 |
| R3 - SO | 1750 | 57.2 |
| R4 - SO | 1815 | 53.5 |

### F.10 ANALYTICAL RESULTS FOR GAS BULB SAMPLES

In the GC/MS analysis of the gas bulb samples, collected from the by-pass line of the hydrocarbon analyses, the analyst was asked to search specifically for the possible presence of chlorinated hydrocarbons. None were detected. However, the composition found for these samples by GC/MS suggest that the gas bulbs had leaked to the extent that the analytical results were not meaningful. This may have been due to unequal coefficients of thermal expansion for Teflon (stopcocks) and glass (bulb) if dramatic temperature changes occurred during sample shipment.

| Constituents | R1(B)-HZ-GB | R3-HZ-GB | R4-HZ-GB |
|---|---|---|---|
| | Concentration, % (v/v) | | |
| Nitrogen | 78+ | 79+ | 78+ |
| Oxygen | 20.8 | 16.5 | 20.7 |
| Argon | 0.93 | 0.93 | 0.94 |
| Carbon Dioxide | 0.042 | 3.1 | 0.12 |
| Hydrogen | <0.005 | <0.005 | <0.005 |
| Methane | <0.005 | <0.005 | <0.005 |
| Chlorinated Hydrocarbons | <0.005 | <0.005 | <0.005 |

# APPENDIX 6-1

# ROLLINS ENVIRONMENTAL SERVICES, INC.
# OPERATING DATA

FOR THE

# THERMAL DESTRUCTION
# OF
# CHEMICAL WASTE

FOR

TRW DEFENSE AND SPACE SYSTEMS GROUP,
TRW, INC.

# CONTENTS

Introduction

Background Test - TRW

Background Test - ADL

Hammermilled Capacitors - TRW

Whole Capacitors - TRW

NCB Test No. 1 - ADL

NCB Test No. 2 - ADL

Cost Estimates

# INTRODUCTION

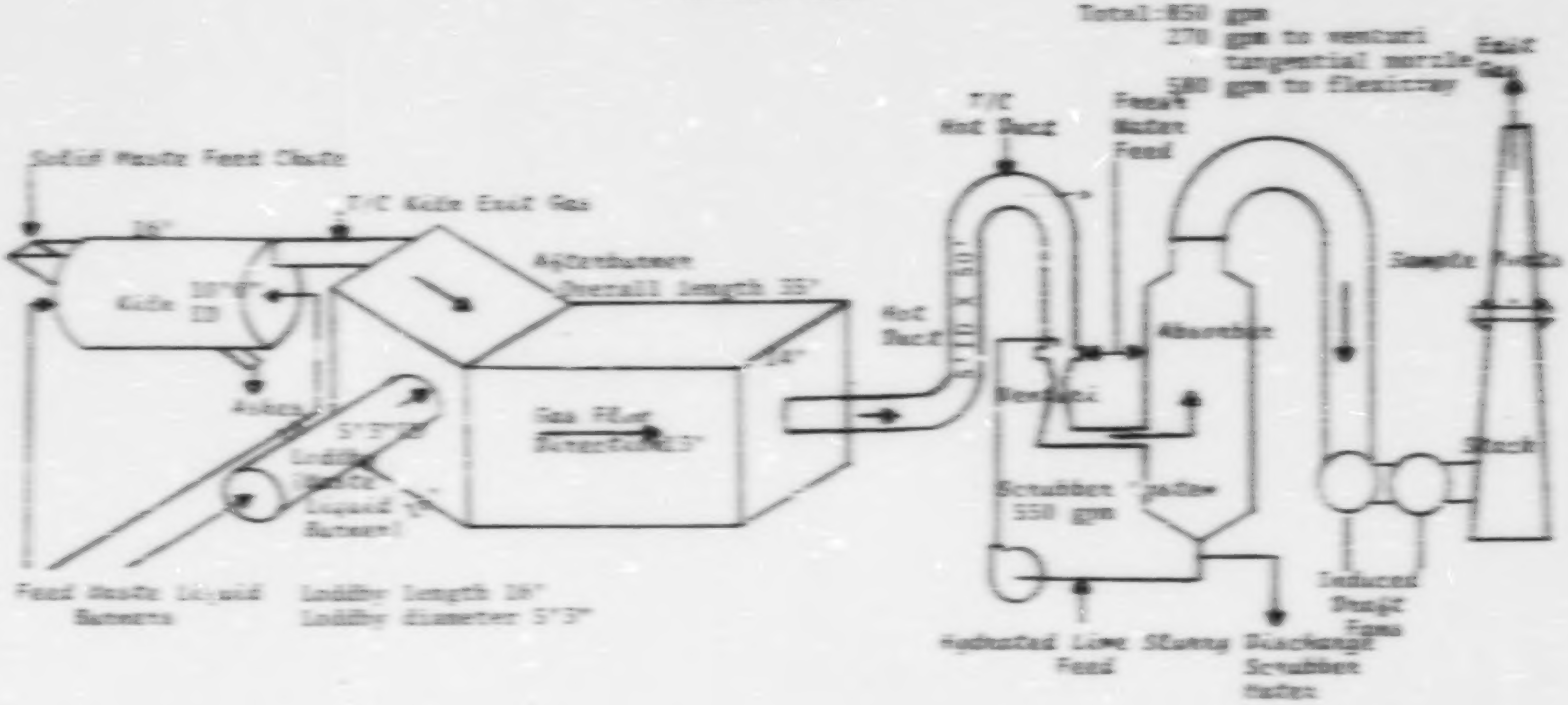
Rollins Environmental Services, Inc. ("RES") operates at its Houston, Texas, facility an industrial waste incinerator capable of thermally destroying liquid, solid and gas wastes. This unit operates at combustion temperatures in excess of 2000°F with a retention time between 2 to 3 seconds. All combustion gases are quenched and scrubbed by a venturi, flexitray scrubber.

This unit, operating 24 hours per day - 7 days per week, is unique in that it has two separate burners feeding a common afterburner. The main burner (loddby) is for waste liquids only and is a vortex type horizontal burner. The second burner is a rotary kiln capable of burning waste liquid, sludges, and solids via fiber packs. Both burners have natural gas ignitors and gas burners for the purpose of initial refractory heat-up, flame stability and supplemental heat, if necessary.

The combustion gases from the above mentioned burners combine in the afterburner with a horizontal cyclone effect. This prolongs the residence time and completes the combustion process prior to the gases leaving the afterburner. From here the gases pass through a duct and enter a wet venturi scrubber, flexitray and demister. The venturi pressure drop is capable of being varied, however, it is normally operated sufficiently so as to scrub all but submicron particles.

Two 400 horsepower induced draft fans in series provide the energy for the scrubber and maintain the entire incinerator at a negative pressure so as to contain all toxic gases. On leaving the fans the gases pass up a one-hundred foot stack to the environment.

ROLLINS ENVIRONMENTAL SERVICES
INCINERATOR SYSTEM CONFIGURATION
HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas
36'
Kiln 10'6" ID
Ashes
Afterburner
Overall Length 35'
24'
Gas Flow Direction
5'3" ID
Loddby Waste Liquid Burner
Feed Waste Liquid Burners
Loddby Length 16'
Loddby Diameter 5'3"
Hot Duct
T/C Hot Duct
Fresh Water Feed
Total:850 gpm
270 gpm to venturi tangential nozzle
580 gpm to Flexitray
Exit Gas
Venturi
Absorber
Sample Ports
Stack
Scrubber Water 550 gpm
Induced Draft Fans
Hydrated Lime Slurry Feed
Discharge Scrubber Water

12/29/76

Destruction testing on two types of waste products was accomplished, the first being polychlorinated biphenyls (PCB) and the second nitrochlorobenzene tars (NCB). Background data was gathered while burning No. 2 oil and then the wastes were burned on subsequent burns using No. 2 oil as the supplementary fuel. The actual conditions of each test will be discussed separately.

## BACKGROUND TEST - TRW

12/06/76

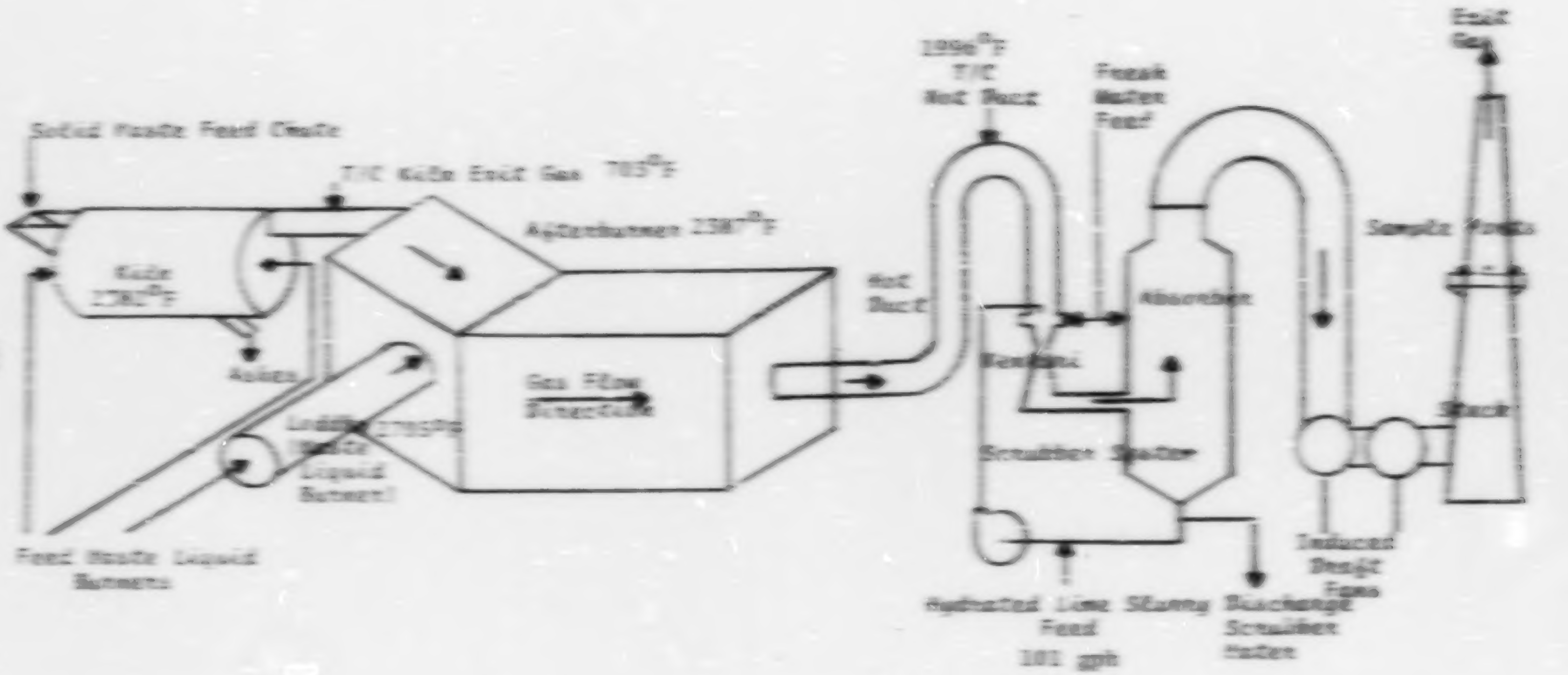
Before all tests a purge burn using No. 2 oil was done for a period of time not less than one hour. This provided for at least six turnovers of scrubber water and sufficient purging of the combustion chambers.

The TRW background test started at 10:45 A.M. with the purge commencing at 8:45 A.M. Both the loddby and kiln waste liquid burners operated on No. 2 fuel oil with a combined feed rate of 552 gallons per hour. Heat release averaged 74.6 million BTU per hour with the average temperatures of the respective points indicated below:

| | |
|---|---|
| Hot duct | 1996°F |
| Kiln duct | 703°F |
| Afterburner | 2387°F |
| Loddby flame | 2705°F |
| Kiln flame | 2382°F |
| Venturi delta P | 41 inches of water |
| Lime consumption | 101 GPH |
| Natural gas consumption | 2095 CFH |

The test concluded at 1400 hours due to severe weather conditions.

ROLLINS ENVIRONMENTAL SERVICES
INCINERATOR SYSTEM CONFIGURATION
HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas 705°F
Afterburner 2387°F
Kiln
2282°F
Ashes
Liquid 2705°F
Waste
Liquid
Burner
Feed Waste Liquid
Burners
Gas Flow
Direction
Hot
Duct
1996°F
T/C
Hot Duct
Fresh
Water
Feed
Venturi
Absorber
Scrubber Water
Sample Ports
Exit
Gas
Stack
Induced
Draft
Fans
Hydrated Lime Slurry
Feed
101 gph
Discharge
Scrubber
Water

12/29/76

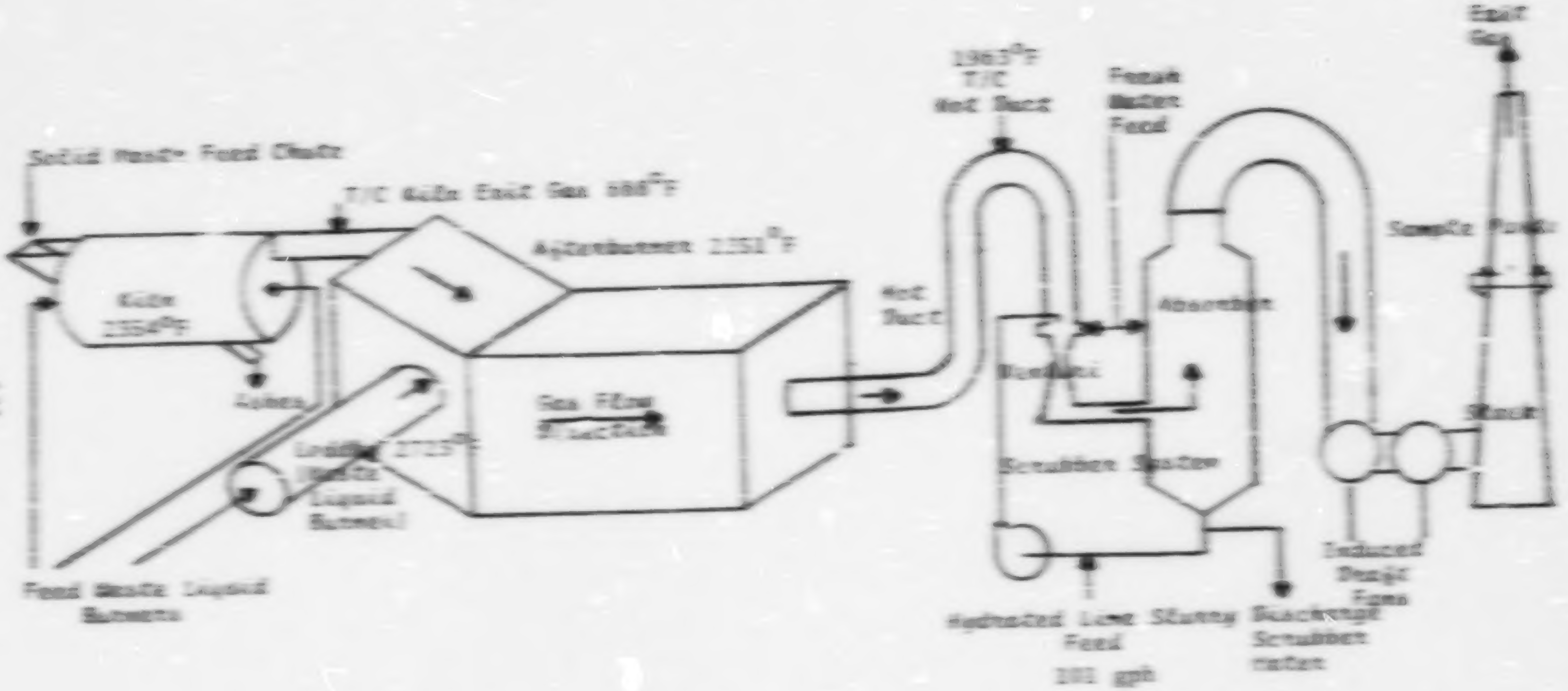
## BACKGROUND TEST - ADL

12/14/76

The oil purge started at 0900 hours and the background test secured at 1630 hours. As before, both the loddby and kiln were fired on diesel fuel, however, the combined feed rate averaged 576 gph with an average heat release of 77.8 million BTU's per hour. The operating data was as follows:

| | |
|---|---|
| Hot duct | 1963°F |
| Kiln duct | 688°F |
| Afterburner | 2351°F |
| Loddby flame | 2723°F |
| Kiln flame | 2354°F |
| Venturi delta P | 36 inches of water |
| Lime consumption | 101 GPH |
| Natural gas consumption | 4923 CFH |
| Retention time | 2.6 seconds |

ROLLINS ENVIRONMENTAL SERVICES
INCINERATOR SYSTEM CONFIGURATION
HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas 880°F
Afterburner 2351°F
Kiln
2354°F
Ashes
Loading 2725°F
Waste
Liquid
Burner
Gas Flow
Direction
Feed Waste Liquid
Burners
Hot
Duct
1963°F
T/C
Hot Duct
Fresh
Water
Feed
Absorber
Venturi
Scrubber System
Hydrated Lime Slurry Feed
101 gph
Discharge
Scrubber
Water
Induced
Draft
Fans
Sample Ports
Stack
Exit
Gas

12/29/76

The test destruction of PCB waste was accomplished burning capacitors in two different states. The first test was conducted with capacitors that were hammermilled and sealed in 55 gallon fiberpacks, and the second test burned whole capacitors.

HAMMERMILLED CAPACITORS - TRW (12/08/76)

In this burn No. 2 oil was fired in the loddby and kiln while the whole fiberpacks were fed into the kiln at a rate varying from one every 4 to 5 minutes. Their weights and feed time are set forth in Figure IV. The total weight of capacitors and fiberpacks was 2558 pounds. As in the background tests, a purge burn was accomplished and started 0830 hours with the test burn commencing at 1000 hours. The test was secured at 1330 hours with a total of 46 fiberpacks burned. The fuel oil burned in the kiln averaged 71 gph or 9.7 million BTU per hour, while the loddby burn rate was 566 gph or 76.5 million BTU per hour. The operating data was as follows:

| | |
|---|---|
| Hot duct | 1993°F |
| Kiln duct | 910°F |
| Afterburner | 2428°F |
| Loddby flame | 2731°F |
| Kiln flame | 2286°F |
| Venturi delta P | 40 inches of water |
| Lime consumption | 101 GPH |
| Natural gas consumption | 3200 CFH |
| Retention time | 3.2 seconds |

## FIGURE IV

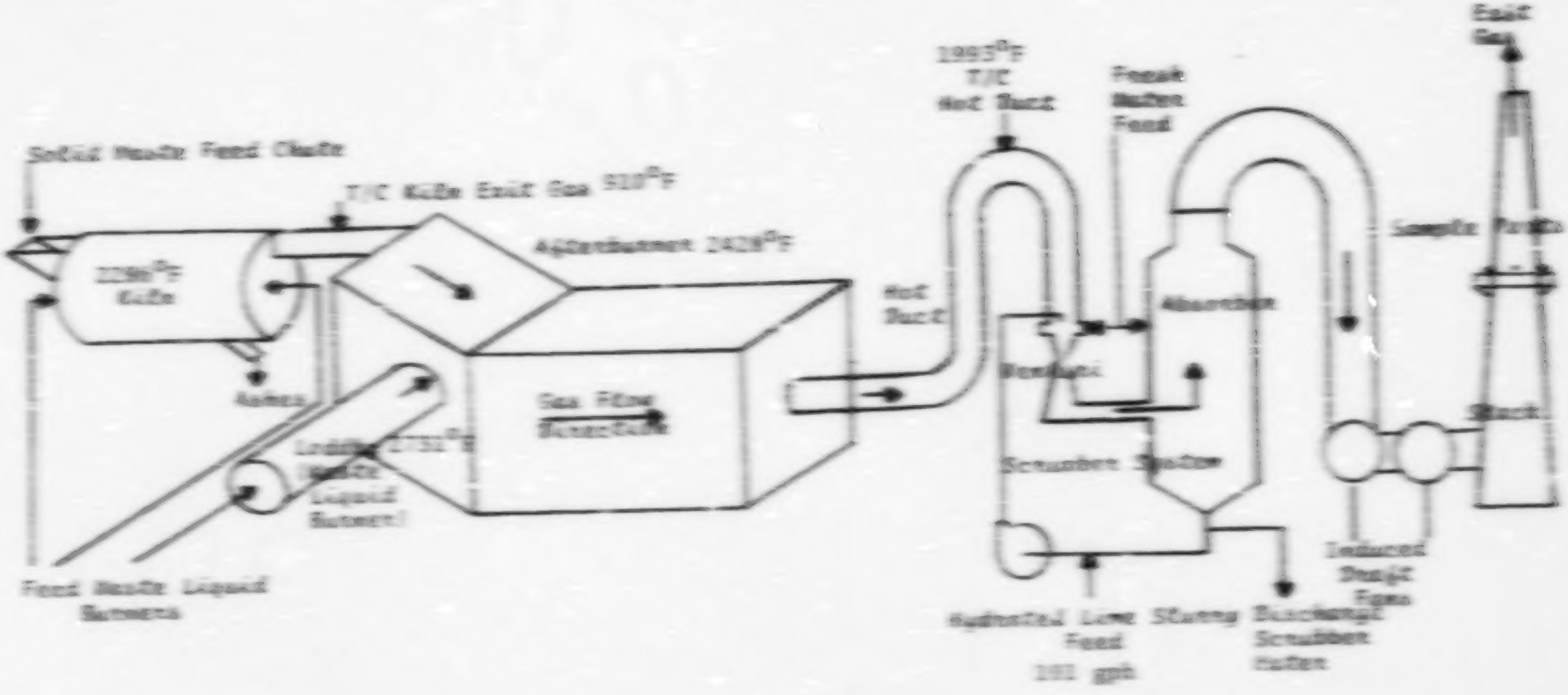
## HAMMERMILLED CAPACITORS

| DRUM NO. | TIME DROP | WEIGHT |
|---|---|---|
| 1 | 0955 | 50 lbs. |
| 2 | 1005 | 52 lbs. |
| 3 | 1010 | 51 lbs. |
| 4 | 1014 | 45 lbs. |
| 5 | 1018 | 68 lbs. |
| 6 | 1022 | 45 lbs. |
| 7 | 1026 | 48 lbs. |
| 8 | 1030 | 81 lbs. |
| 9 | 1034 | 67 lbs. |
| 10 | 1038 | 67 lbs. |
| 11 | 1042 | 50 lbs. |
| 12 | 1046 | 46 lbs. |
| 13 | 1050 | 51 lbs. |
| 14 | 1054 | 75 lbs. |
| 15 | 1100 | 52 lbs. |
| 16 | 1204 | 50 lbs. |
| 17 | 1108 | 56 lbs. |
| 18 | 1112 | 49 lbs. |
| 19 | 1116 | 56 lbs. |
| 20 | 1120 | 75 lbs. |
| 21 | 1124 | 65 lbs. |
| 22 | 1128 | 47 lbs. |
| 23 | 1132 | 55 lbs. |
| 24 | 1136 | 69 lbs. |
| 25 | 1140 | 44 lbs. |
| 26 | 1144 | 46 lbs. |
| 27 | 1148 | 47 lbs. |
| 28 | 1152 | 46 lbs. |
| 29 | 1156 | 43 lbs. |
| 30 | 1200 | 43 lbs. |

## FIGURE IV
## HAMMERMILLED CAPACITORS: CONTINUED

| DRUM NO. | TIME DROP | WEIGHT |
|---|---|---|
| 31 | 1204 | 47 lbs. |
| 32 | 1208 | 56 lbs. |
| 33 | 1212 | 63 lbs. |
| 34 | 1216 | 61 lbs. |
| 35 | 1220 | 67 lbs. |
| 36 | 1225 | 56 lbs. |
| 37 | 1240 | 53 lbs. |
| 38 | 1245 | 45 lbs. |
| 39 | 1250 | 80 lbs. |
| 40 | 1255 | 50 lbs. |
| 41 | 1300 | 43 lbs. |
| 42 | 1305 | 53 lbs. |
| 43 | 1310 | 40 lbs. |
| 44 | 1315 | 78 lbs. |
| 45 | 1320 | 70 lbs. |
| 46 | 1325 | 57 lbs. |

ROLLINS ENVIRONMENTAL SERVICES
INCINERATOR SYSTEM CONFIGURATION
(HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas 910°F
2290°F
Kiln
Afterburner 2428°F
Ashes
Gas Flow
Direction
Loading 2731°F
(Waste
Liquid
Burner)
Feed Waste Liquid
Burners
Hot
Duct
1993°F
T/C
Hot Duct
Fresh
Water
Feed
Venturi
Absorber
Scrubber System
Hydrated Lime Slurry
Feed
101 gph
Discharge
Scrubber
Water
Induced
Draft
Fans
Sample Ports
Stack
Exit
Gas

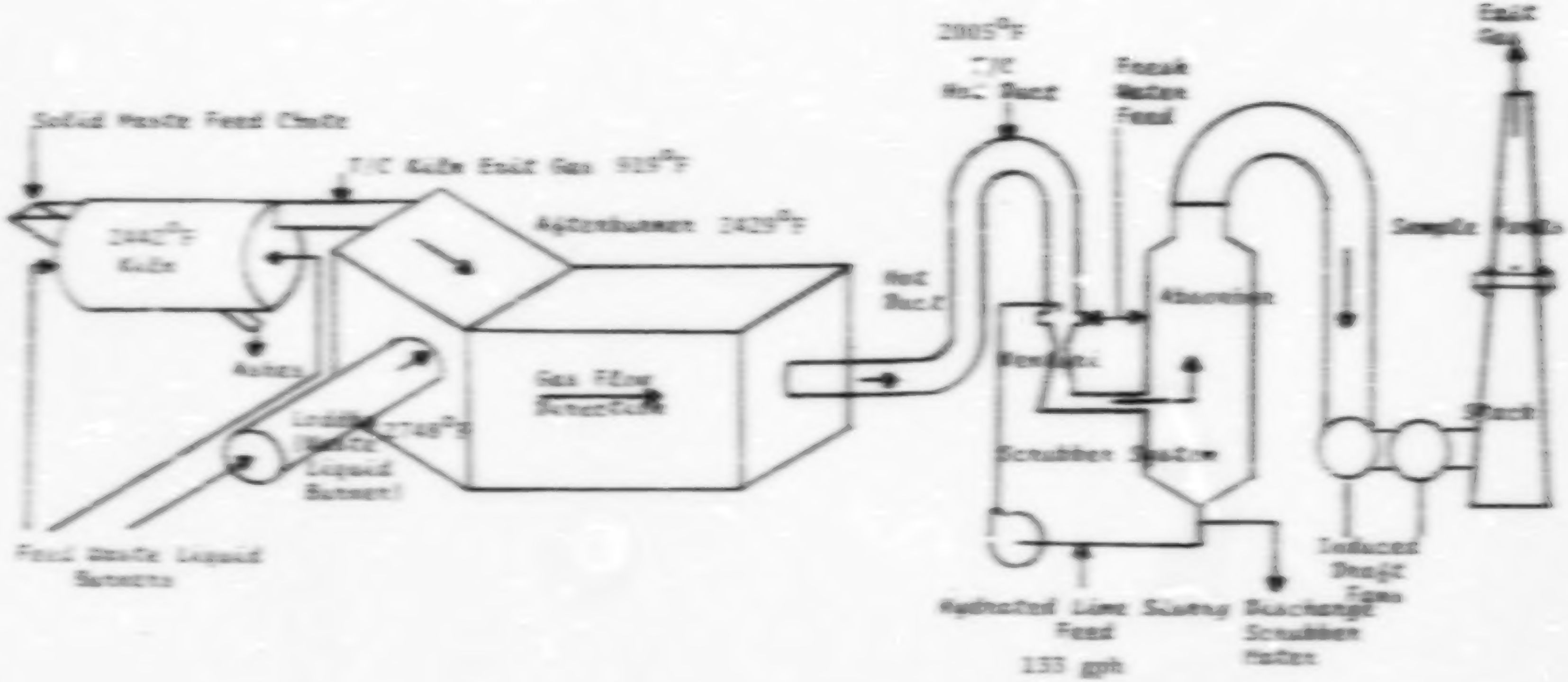
12/29/76

## WHOLE CAPACITORS - TRW (12/09/76)

As was in the case of the hammermilled capacitors, fuel oil was burned in the kiln and loddby while whole capacitors were fed evenly as possible over a 5 hour-15 minute test period. Purge started at 1045 hours with capacitors starting at 1200 hours and ending at 1515 hours. Six drums of capacitors were fed with the average weight being approximately 450 pounds per drum. Feed rates to the loddby averaged 460 pgh or 62.2 million BTU per hour while the kiln averaged 148 gph or 20 million BTU per hours. Operating data was as follows:

| | |
|---|---|
| Hot duct | 2005°F |
| Kiln duct | 910°F |
| Afterburner | 2429°F |
| Loddby flame | 2748°F |
| Kiln flame | 2442°F |
| Venturi delta P | 40 inches of water |
| Lime consumption | 135 GPH |
| Natural gas consumption | 3100 CFH |
| Retention time | 3.0 seconds |

ROLLINS ENVIRONMENTAL SERVICES
INCINERATOR SYSTEM CONFIGURATION
HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas 919°F
2442°F Kiln
Ashes
Afterburner 2429°F
Liquid Waste Liquid Burner 2748°F
Gas Flow Direction
Feed Waste Liquid Burners
Hot Duct
2005°F T/C Hot Duct
Fresh Water Feed
Venturi
Absorber
Scrubber System
Hydrated Lime Slurry Feed 133 gph
Discharge Scrubber Water
Induced Draft Fans
Sample Ports
Stack
Exit Gas

12/09/76

The destruction of NCB tars was tested twice under similar conditions except that the hot duct temperature was lowered for the second test. The NCB tars were diluted with No. 2 oil to a blend of 20% NCB and 80% fuel oil by volume. Analysis of this blend was as follows:

| | |
|---|---|
| BTU per pound | 17584 |
| Pounds per gallon | 7.88 |
| BTU per gallon | 138,562 |
| Scrub pound per pound | .156 |
| Chlorine | 10.2% by wt. |
| Ash | 0.8% by wt. |

NCB TEST NO. 1 - ADL (12/17/76)

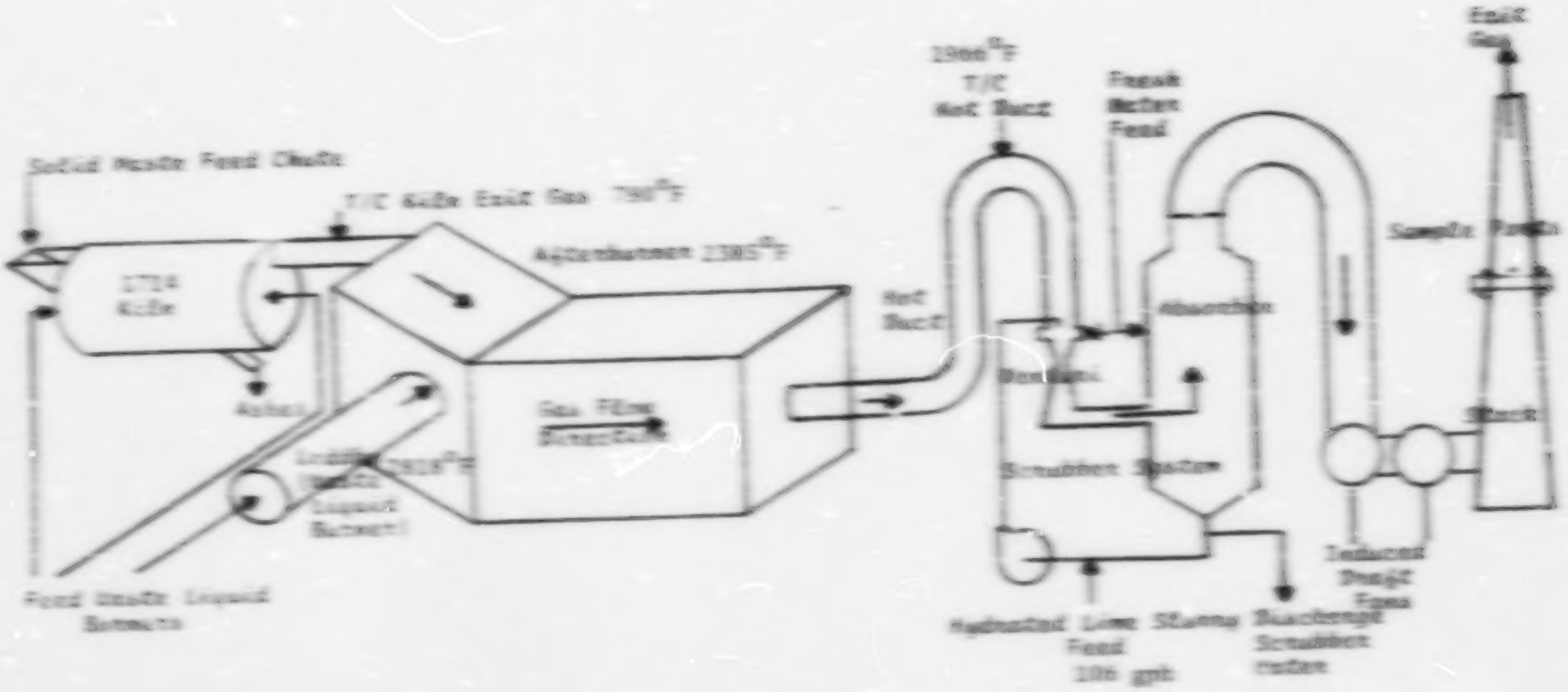
This test started its purge at 0909 hours with the sampling commencing at 1045 hours and ending at 1400 hours. The kiln was fed No. 2 oil at a feed rate of 65.8 gph or 8.0 million BTU per hour while the loddby was fed the blend at 534 gph or 74 million BTU per hour. The pounds per hour of NCB burned in the loddby averaged 1099. The operating data was as follows:

| | |
|---|---|
| Hot duct | 1966°F |
| Kiln duct | 700°F |
| Afterburner | 2385°F |
| Loddby flame | 2618°F |
| Kiln flame | 1714°F |
| Venturi delta P | 43 inches of water |
| Lime consumption | 106 GPH |
| Natural gas consumption | 4065 CFH |
| Retention time | 2.33 seconds |

NCB TEST NO. 2 - ADL (12/18/76)

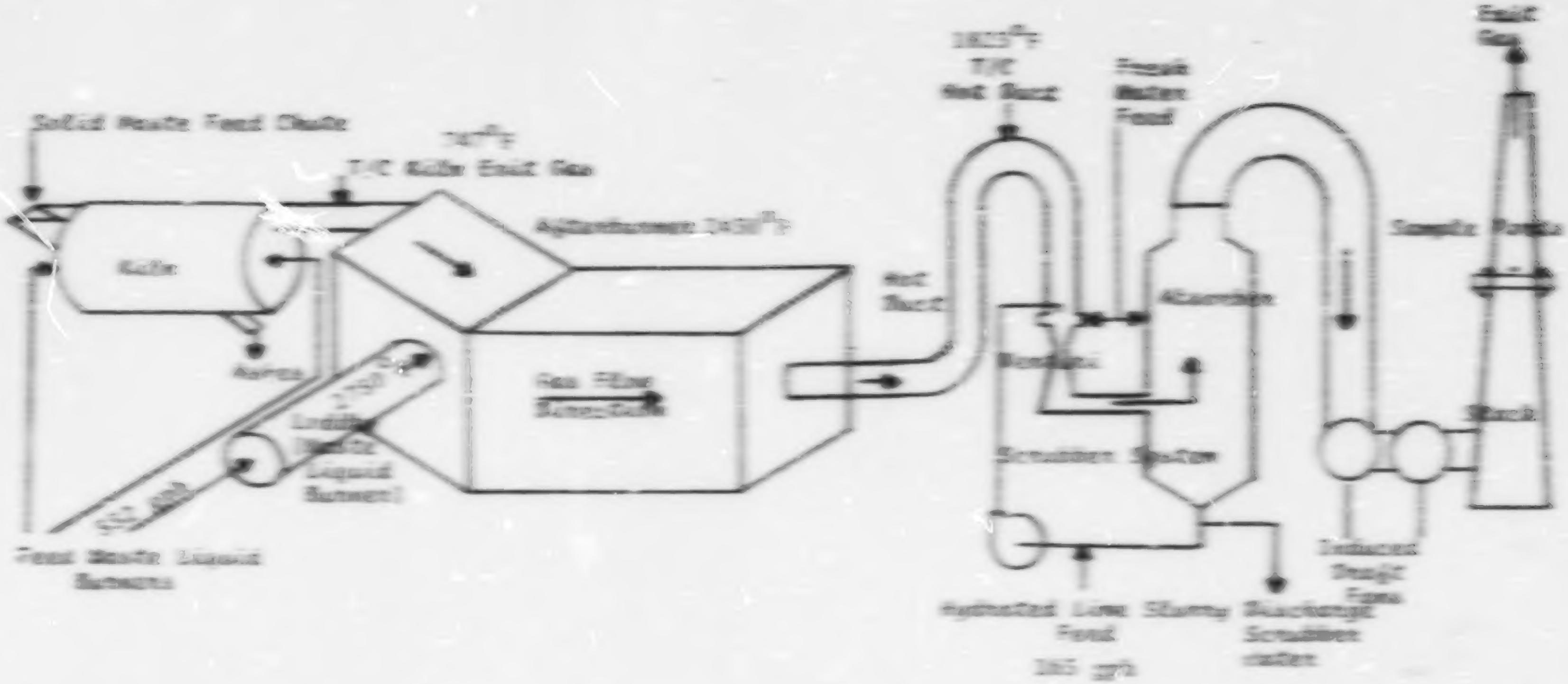
The purge started at 0930 hours and testing began at 1030 hours and finished at 1530 hours. The same blend was fed in the loddby and No. 2 oil again was burned in the kiln. The loddby feed rate was

### ROLLINS ENVIRONMENTAL SERVICES
### INCINERATOR SYSTEM CONFIGURATION
### HOUSTON PLANT


Solid Waste Feed Chute
T/C Kiln Exit Gas 790°F
Afterburner 2305°F
Kiln
Ashes
Gas Flow Direction
Liquid Burner
Feed Waste Liquid Burners
Hot Duct
1900°F T/C Hot Duct
Fresh Water Feed
Venturi
Absorber
Scrubber System
Hydrated Lime Slurry Feed 106 gph
Discharge Scrubber Water
Induced Draft Fans
Sample Ports
Stack
Exit Gas

12/29/76

# ROLLINS ENVIRONMENTAL SERVICES
# INCINERATOR SYSTEM CONFIGURATION
# HOUSTON PLANT


Solid Waste Feed Chute
747°F
T/C Kiln Exit Gas
Afterburner 2450°F
Kiln
Aprons
Gas Flow Direction
Liquid Waste Liquid Burner
Feed Waste Liquid Burners
Hot Duct
1825°F
T/C
Hot Duct
Fresh Water Feed
Venturi
Absorber
Scrubber System
Hydrated Lime Slurry Feed
165 gph
Discharge Scrubber Water
Induced Draft Fans
Sample Ports
Stack
Exit Gas

[illegible]

466 gph or 64.6 million BTU per hour, while the kiln was 104 gph or 14 million BTU per hour. The above loddby feed rate gave an average input of 959 pounds per hour of NCB to the loddby. The operating date was as follows:

| | |
|---|---|
| Hot duct | 1823°F |
| Kiln duct | 747°F |
| Afterburner | 2450°F |
| Loddby flame | 2750°F |
| Kiln flame | Not available |
| Venturi delta P | 43 inches of water |
| Lime consumption | 165 GPH |
| Natural gas consumption | 3500 CFH |
| Retention time | 2.31 seconds |

## COST ESTIMATES

Capital Investment Needed
for Turnkey installation
including land, tanks, scrubber ponds,
utilities and incineration system — $5,000,000

Operating personnel requirements
1 Supervisor per shift 7 days per week
3 operators per shift 7 days per week
1 lab technician 2 shifts per day 7 days
per week
4 mechanics one shift per day
5 days per week
Levels of skill include: pipefitting
machinist
welder
millwright

Approximate estimate of yearly maintenance expense
less labor — $ 196,000

Approximate estimate of yearly maintenance labor other
than in house- i.e. refractory, fiberglass, and electrical
$ 85,000

# APPENDIX G-2
# INCINERATION SYSTEM RAW DATA

## TABLE 6-[illegible]

## TEST I - [illegible] BACKGROUND

| Date [illegible] Time | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Afterburner Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |  | [illegible] |
| [illegible] Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |  | [illegible] |
| Forced Draft Fan (Amps) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Induced Draft Fan (Amps) | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] |
| Delta-P Venturi (In. $H_2O$) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Gas Meter Reading | [illegible] |  |  |  |  |  |  |  | [illegible] |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |

## TABLE G-1

## TEST I - TNW BACKGROUND

(continued)

| Date: 12/6/78 Time | 1145 | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 | 1345 | 1400 | 1415 | 1430 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 2000 | 1975 | 2000 | 2000 | 2050 | 2025 | 2000 | 1975 | 2000 | 1950 | | |
| Kiln Temp (°F) | 750 | 700 | 700 | 700 | 750 | 750 | 600 | 600 | 700 | 700 | | |
| Afterburner Temp (°F) (actual) | 2420 | 2410 | 2400 | 2422 | 2420 | 2420 | 2400 | 2364 | 2410 | 2400 | | |
| Loading Flame Temp (°F) Optical | 2720 | 2710 | 2700 | 2710 | 2720 | 2720 | 2700 | 2710 | 2700 | 2680 | | |
| Kiln Flame Temp (°F) Optical | 2310 | 2330 | 2400 | 2290 | 2380 | 2400 | 2390 | 2400 | 2400 | 2370 | | |
| Forced Draft Fan (Amps) | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | | |
| Induced Draft Fan (Amps) | 4.2 / 450 | 420 / 460 | 420 / 450 | 420 / 460 | 420 / 364 | 420 / 460 | 410 / 475 | 420 / 460 | 410 / 460 | 420 / 460 | | |
| Delta-P Venturi (in. $H_2O$) | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | | |
| Gas Meter Reading | | | 132844 | | | | | | | 133007 | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE 6-2

## TEST II - HAMMERMILLED PCB CAPACITORS

| Date [illegible] Time | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Afterburner Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| [illegible] Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Forced Draft Fan (Amps) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Induced Draft Fan (Amps) | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] | [illegible] / [illegible] |
| Delta-P Venturi (In. $H_2O$) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Gas Meter Reading | [illegible] | | | [illegible] | | | [illegible] | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE G-2

## TEST II - HAMMERMILLED PCB CAPACITORS
(continued)

| Date: 12/8/76 Time | 1130 | 1145 | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 | 1345 | 1400 | 1415 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 2100 | 1975 | 2000 | 2000 | 2000 | 2000 | 2000 | 2000 | 2000 | 1950 | | |
| Kiln Temp (°F) | 900 | 850 | 900 | 850 | 900 | 775 | 1000 | 900 | 950 | 1000 | | |
| Afterburner Temp (°F) Optical | 2420 | 2400 | 2411 | 2400 | 2420 | 2450 | 2420 | 2410 | 2450 | 2400 | | |
| Lobby Flame Temp (°F) Optical | 2740 | 2775 | 2710 | 2720 | 2750 | 2740 | 2710 | 2730 | 2720 | 2720 | | |
| Kiln Flame Temp (°F) Optical | 2740 | 2700 | 2720 | 2700 | 2720 | 2720 | 2750 | 2770 | 2730 | - | | |
| Forced Draft Fan (Amps) | 61 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | | |
| Induced Draft Fan (Amps) | 430 / 460 | 430 / 460 | 425 / 450 | 415 / 445 | 41x / 456 | 425 / 455 | 422 / 447 | 430 / 455 | 430 / 460 | 432 / 460 | | |
| C-Ring-P Venturi (In. $H_2O$) | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | | |
| Gas Meter Reading | | | | | | | | | [illegible] | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE 6-3

## TEST III - WHOLE PCB CAPACITORS

| Date [illegible] Time | 1045 | 1100 | 1115 | 1130 | 1145 | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp ($^{o}F$) | 1880 | 1950 | 1985 | 2075 | 1940 | 1900 | 1975 | 1960 | 2050 | 2050 | 1950 | 2090 |
| Kiln Temp ($^{o}F$) | 950 | 1025 | 1000 | 1050 | 1100 | 1000 | 1000 | 950 | 975 | 975 | 980 | 980 |
| Afterburner Temp ($^{o}F$) Optical | 2330 | 2380 | 2370 | 2570 | 2400 | 2340 | 2420 | 2330 | 2530 | 2540 | 2420 | 2400 |
| Lobby Flame Temp ($^{o}F$) Optical | 2715 | 2730 | 2780 | 2790 | 2720 | 2740 | 2700 | 2710 | 2790 | 2770 | 2750 | 2740 |
| Kiln Flame Temp ($^{o}F$) Optical | 2400 | 2470 | 2470 | 2570 | 2450 | 2390 | 2370 | 2420 | 2460 | 2460 | 2410 | 2370 |
| Forced Draft Fan (Amps) | 62 | 61 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | [illegible] | 62 |
| Induced Draft Fan (Amps) | 450 / 460 | 455 / 455 | 455 / 455 | 450 / 455 | 450 / 455 | 455 / 455 | 455 / 455 | 455 / 455 | 440 / 450 | 450 / 455 | 440 / 460 | 450 / 460 |
| Delta-P Venturi (in. $H_2O$) | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" | 40" |
| Gas Meter Reading | 194332 | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

TABLE 6-3

TEST III - WHOLE PCB CAPACITORS
(continued)

| Date 12/9/76 Time | 1345 | 1400 | 1415 | 1430 | 1445 | 1500 | 1515 | 1530 | 1545 | 1600 | 1615 | 1630 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 2550 | 2550 | 2580 | 2600 | 1950 | 2590 | 2550 | | | | | |
| Kiln Temp (°F) | 900 | 900 | 900 | 850 | 825 | 850 | 850 | | | | | |
| Afterburner Temp (°F) Optical | 2420 | 2440 | 2440 | 2430 | 2400 | 2450 | 2450 | | | | | |
| Lobby Flame Temp (°F) Optical | 2750 | 2760 | 2770 | 2770 | 2750 | 2730 | 2750 | | | | | |
| Kiln Flame Temp (°F) Optical | 2460 | 2480 | 2470 | 2410 / 2450 | 2420 | 2410 | 2480 | | | | | |
| Forced Draft Fan (Amps) | 62 | 62 | 62 | 62 | 62 | 62 | 62 | | | | | |
| Induced Draft Fan (Amps) | 420 / 460 | 420 / 460 | 420 / 460 | 420 / 460 | 415 / 460 | 415 / 440 | 420 / 460 | | | | | |
| Delta-P Venturi (in. Hg) | 40" | 40" | 40" | 42" | 40" | 40" | 42" | | | | | |
| Gas Meter Reading | | | | | | | 78459 | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

# TABLE 6-4

## RUN #7(B) BACKGROUND TEST

| Date: 12/14/76 Time | 1600 | 1615 | 1630 | 1645 | 1700 | 1715 | 1730 | 1745 | 1800 | 1815 | 1830 | 1845 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 2050 | 2000 | 2000 | 1950 | 1950 | 1950 | 1925 | 1975 | 2000 | 1960 | 1940 | 1950 |
| Kiln Temp (°F) | 650 | 650 | 650 | 600 | 575 | 575 | 600 | 625 | 600 | 600 | 650 | 600 |
| Afterburner Temp (°F) Optical | 2400 | 2350 | 2400 | 2425 | 2400 | 2400 | 2360 | 2360 | 2360 | 2350 | 2350 | 2360 |
| Lobby Flame Temp (°F) Optical | 2700 | 2750 | 2750 | 2710 | 2725 | 2725 | 2725 | 2730 | 2725 | 2730 | 2730 | 2710 |
| Kiln Flame Temp (°F) Optical | 2400 | 2425 | 2400 | 2385 | 2350 | 2350 | 2350 | 2375 | 2356 | 2380 | 2340 | 2320 |
| Forced Draft Fan (Amps) | 61 | 62 | 62 | 63 | 63 | 63 | 63 | 63 | 63 | 63 | 63 | 63 |
| Induced Draft Fan (Amps) | 425 / 425 | 425 / 425 | 425 / 425 | 425 / 425 | 425 / 425 | 400 / 425 | 400 / 425 | 427 / 425 | 427 / 425 | 407 / 425 | 425 / 425 | 425 / 425 |
| Delta-P Venturi (In. H₂O) | 35" | 35" | 35" | 35" | 35" | 35" | 35" | 35" | 35" | 35" | 38" | 38" |
| Gas Meter Reading | 14600 | START | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE G-4

## RUN R7(B) BACKGROUND TEST

(continued)

| Date 12/14/76 Time | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 | 1345 | 1400 | 1415 | 1430 | 1445 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 1897 | 1950 | 1950 | 1925 | 1900 | 1925 | 1950 | 1960 | 1960 | 1950 | 1975 | 2000 |
| Kiln Temp (°F) | 650 | 650 | 650 | 650 | 625 | 650 | 650 | 750 | 750 | 750 | 750 | 800 |
| Afterburner Temp (°F) Optical | 2310 | 2360 | 2360 | 2220 | 2290 | 2330 | 2350 | 2340 | 2370 | 2330 | 2350 | 2330 |
| Lobby Flame Temp (°F) Optical | 2720 | 2740 | 2740 | 2700 | 2600 | 2720 | 2720 | 2720 | 2750 | 2750 | 2730 | 2730 |
| Kiln Flame Temp (°F) Optical | 2350 | 2350 | 2350 | 2330 | 2370 | 2340 | 2350 | 2350 | 2370 | 2330 | 2340 | 2400 |
| Forced Draft Fan (Amps) | 63 | 63 | 63 | 63 | 63 | 63 | 63 | 63 | 62 | 62 | 62 | 62 |
| Induced Draft Fan (Amps) | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 430 / 430 | 425 / 425 | 425 / 425 |
| Delta-P Venturi (In. $H_2O$) | 30" | 30" | 30" | 30" | 30" | 30" | 30" | 30" | 30" | 30" | 30" | 30" |
| Gas Meter Reading | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

TABLE G-4

RDP RT(8) BACKGROUND TEST

(continued)

| Date 12/14/76 Time | 1500 | 1515 | 1530 | 1545 | 1600 | 1615 | END TEST 1630 | 1645 | 1700 | 1715 | 1730 | 1745 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 2000 | 2000 | 2000 | 1975 | 1975 | 1950 | 1950 | | | | | |
| Kiln Temp (°F) | 800 | 800 | 600 | 800 | 900 | 750 | 750 | | | | | |
| Afterburner Temp (°F) Optical | 2230 | 2230 | 2230 | 2250 | 2250 | 2250 | 2250 | | | | | |
| Lobby Flame Temp (°F) Optical | 2710 | 2710 | 2720 | 2730 | 2720 | 2720 | 2720 | | | | | |
| Kiln Flame Temp (°F) Optical | 2400 | 2400 | 2300 | 2360 | 2350 | 2330 | 2330 | | | | | |
| Forced Draft Fan (Amps) | 42 | 42 | 42 | 42 | 42 | 42 | 42 | | | | | |
| Induced Draft Fan (Amps) | 420 / 420 | 420 / 420 | 420 / 420 | 420 / 430 | 420 / 430 | 420 / 430 | 420 / 430 | | | | | |
| Delta-P Venturi (In. $H_2O$) | 30" | 30" | 30" | 30" | 30" | 30" | 30" | | | | | |
| Gas Meter Reading | | | | | | | [illegible]<br>[illegible] | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE G-5

## RUN #2 – NCB TO KILN

| Date 12/15/76 Time | 0900 | 0915 | 0930 | 0945 | 1000 | 1015 | 1030 | 1045 | 1100 | 1115 | 1130 | 1145 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Wet Duct Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Temp (°F) | [illegible] | [illegible] | 700 | 750 | 750 | 750 | 750 | 750 | 750 | [illegible] | 775 | 775 |
| Afterburner Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Lubby Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Forced Draft Fan (Amps) | [illegible] | [illegible] | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 | 62 |
| Induced Draft Fan (Amps) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Delta-P Venturi (In. $H_2O$) | 39" | 39" | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Lubby Feed Tank Level (Inches)** | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Gas Meter Reading | [illegible] | [illegible] | | | | | | | | | | |
| | | | | | | | | | | | | |

** 1 inch – 60 gallons

## TABLE 6-5
## RUN [illegible] – [illegible] TO KILN
(continued)

| Date [illegible] Time | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 | 1345 | 1400 | 1415 | 1430 | [illegible] |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Temp (°F) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Afterburner Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| [illegible] Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Kiln Flame Temp (°F) Optical | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Forced Draft Fan (Amps) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Induced Draft Fan (Amps) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Delta-P Venturi (in. Hg) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| [illegible] Feed Tank Level (Inches)* | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE 6-5

### RUN R2 – NCB TO KILN
(continued)

| Date 12/15/78 Time | 1500 | 1515 | 1530 | END TEST 1540 | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 1900 | 1900 | 1900 | 1900 | | | | | | | | |
| Kiln Temp (°F) | 590 | 590 | 590 | 590 | | | | | | | | |
| Afterburner Temp (°F) Optical | 2330 | 2330 | 2320 | 2300 | | | | | | | | |
| Lobby Flame Temp (°F) Optical | 2720 | 2710 | 2710 | 2700 | | | | | | | | |
| Kiln Flame Temp (°F) Optical | 2220 | 2200 | 2170 | 2150 | | | | | | | | |
| Forced Draft Fan (Amps) | 67 | 67 | 67 | 67 | | | | | | | | |
| Induced Draft Fan (Amps) | 460 / 420 | 460 / 420 | 460 / 420 | 460 / 420 | | | | | | | | |
| Delta-P Venturi (In. $H_2O$) | 47" | 47" | 47" | 47" | | | | | | | | |
| Lobby Feed Tank Level (Inches) | [illegible] | [illegible] | [illegible] | [illegible] | | | | | | | | |
| Gas Meter Reading | | | | [illegible]<br>[illegible] | | | | | | | | |
| | | | | | | | | | | | | |

## TABLE 6-6

## RUN #3 - NCB - DIESEL FUEL TO LOBBY

| Date 12/7/78 Time | 0845 | 0900 | 0915 | 0930 | 0945 | 1000 | 1015 | 1030 | 1045 | 1100 | 1115 | 1130 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | | 1960 | 2150 | 1950 | 1960 | 1950 | 1960 | 1950 | 1975 | 1975 | 1975 | 2000 |
| Kiln Temp (°F) | | 800 | 800 | 750 | 710 | 750 | 800 | 825 | 800 | 800 | 775 | 790 |
| Afterburner Temp (°F) (Optical) | | 2400 | 2600 | 2350 | 2450 | 2400 | 2450 | 2450 | 2470 | 2400 | 2400 | 2440 |
| Lobby Flame Temp (°F) (Optical) | | 2640 | 2750 | 2700 | 2740 | 2790 | 2840 | 2800 | 2850 | 2800 | 2800 | 2820 |
| Kiln Flame Temp (°F) (Optical) | | | | | | | | | 2370 | 2350 | 2380 | 2400 |
| Forced Draft Fan (Amps) | | | | | | | | | | | | |
| Induced Draft Fan (Amps) | | 385-410<br>300-460 | 385-410<br>300-460 | 385-410<br>300-460 | 385-410<br>300-460 | 385-410<br>300-460 | 385-410<br>300-460 | 385-410<br>300-460 | 385-420<br>300-460 | 385-420<br>300-460 | 385-420<br>300-460 | 385-420<br>300-460 |
| Delta-P Venturi (in. $H_2O$) | | 42" | 42" | 44" | 42" | 42" | 42" | 42" | 42" | 42" | | |
| Lobby Feed Tank (Level) (36,000 gal = 22,900 gal)* | 36" | 37" | 38" | 42" | 44" | 45" | 46" | 48" | 50" | 52" | 54" | 56" |
| Lime Feed Tank Level (Inches)** | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 44" |
| Gas Meter Reading | 23987<br>74032 | | | | | | | | | | | |

* 1 inch = 69 gallons

** 20% $Ca(OH)_2$ by wt., 1 inch = 110 gallons

## TABLE 6-6

## RUN #3 - NCB - DIESEL FUEL TO LOBBY
(continued)

| Date 12/1/76 Time | 1145 | 1200 | 1215 | 1230 | 1245 | 1300 | 1315 | 1330 | 1345 | 1400 | 1415 | 1430 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 1950 | 1960 | 1960 | 1960 | 1960 | 1950 | 1960 | 1960 | 2000 | 2000 | | |
| Kiln Temp (°F) | 790 | 790 | 790 | 790 | 790 | 790 | 790 | 790 | 790 | 790 | | |
| Afterburner Temp (°F) Optical | 2380 | 2440 | 2410 | 2440 | 2420 | 2400 | 2360 | 2340 | 2290 | 2300 | | |
| Lobby Flame Temp (°F) Optical | 2790 | 2900 | 2910 | 2850 | 2900 | 2840 | 2780 | 2770 | 2770 | 2800 | | |
| Kiln Flame Temp (°F) Optical | 2300 | 2300 | 2230 | 2300 | 2300 | 2300 | 2300 | 2300 | 2310 | 2310 | | |
| Forced Draft Fan (Amps) | | | | | | | | | | | | |
| Induced Draft Fan (Amps) | 390 430<br>392 460 | 390 430<br>392 460 | 390 430<br>392 460 | 390 410<br>392 460 | 390 430<br>392 460 | 390 410<br>392 460 | 390 410<br>392 460 | 390 410<br>392 460 | 390 410<br>392 460 | 390 410<br>392 460 | | |
| Delta-P Venturi (In. $H_2O$) | 42" | 42" | 42" | 42" | 42" | 42" | 44" | 44" | 44" | 44" | | |
| Lobby Feed Tank Level (In.) DIST. #2 F.O.* | 54" | 59" | 65" | 67" | 64" | 67" | 70" | 72" | 72" | 74" | 76" | |
| Lime Feed Tank Level (Inches)** | 48" | 48" | 48" | 48" | 48" | 48" | 48" | 48" | 48" | 48" | 48" | |
| Gas Meter Reading | | | | | | | | | | | STOP 788854 | |

* 1 inch - 42 gallons

** 20% $Ca(OH)_2$ by wt., 1 inch - 170 gallons

## TABLE 6-7

## RUN R4 - MCB - DIESEL FUEL TO LODDBY

| Date 12/02/76 Time | 0930 | 0945 | 1000 | 1015 | 1030 | 1045 | 1100 | 1115 | 1130 | 1145 | 1200 | 1215 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 1725 | 1900 | 1750 | 1900 | 1825 | 1900 | 1800 | 1925 | 1800 | 1850 | 1850 | 1875 |
| Kiln Temp (°F) | 750 | 800 | 700 | 800 | 740 | 750 | 700 | 700 | 710 | 790 | 800 | 800 |
| Afterburner Temp (°F) Optical | 2130 | 2310 | 2270 | 2330 | 2350 | 2350 | 2330 | 2400 | 2420 | 2460 | 2450 | 2430 |
| Loddby Flame Temp (°F) Optical | 2670 | 2700 | 2660 | 2700 | 2620 | 2700 | 2720 | 2710 | 2740 | 2760 | 2780 | 2780 |
| Kiln Flame Temp (°F) Optical | | | | | | | | | | | | |
| Forced Draft Fan (Amps) | | | | | | | | | | | | |
| Induced Draft Fan (Amps) | 420 / 450 | 440 / 470 | 430 / 460 | 420 / 450 | 410 / 460 | 430 / 460 | 420 / 460 | 430 / 460 | 430 / 460 | 420 / 460 | 420 / 460 | 410 / 460 |
| Delta-P Venturi (In. $H_2O$) | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" | 42" |
| Loddby Feed Tank (Diesel) Level (In.) [illegible] MCB * | 75" | 77" | 79" | 80" | 81" | 83" | 85" | 87" | 88" | 90" | 92" | 96" |
| Lime Feed Tank Level (Inches)** | 42" | 42" | 42" | 42" | 44" | 44" | 44" | 44" | 45" | 45" | 45" | 46" |
| Gas Meter Reading | [illegible] | | | | | | | | | | | |

* 1 inch - 44 gallons

** 20% $Ca(OH)_2$ by wt., 1 inch - 110 gallons

## TABLE 6-7

## RUN R4 - NCB - DIESEL FUEL TO LODDBY
(continued)

| Date [illegible] Time | 1224 | 1245 | 1300 | 1315 | 1330 | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Hot Duct Temp (°F) | 1900 | 1790 | 1590 | 1850 | 1750 | | | | | | | |
| Kiln Temp (°F) | 800 | 790 | 720 | 780 | 770 | | | | | | | |
| Afterburner Temp (°F) Optical | 2500 | 2500 | 2450 | 2470 | 2420 | | | | | | | |
| Loddby Flame Temp (°F) Optical | 2900 | 2900 | 2440 | 2960 | 2900 | | | | | | | |
| Kiln Flame Temp (°F) Optical | | | | | | | | | | | | |
| Forced Draft Fan (Amps) | | | | | | | | | | | | |
| Induced Draft Fan (Amps) | 410 / 460 | 410 / 460 | 410 / 460 | 410 / 460 | 411 / 460 | | | | | | | |
| Delta-P Venturi (In. $H_2O$) | 42" | 42" | 42" | 42" | 40" | | | | | | | |
| Loddby Feed Tank Blend Level (In.) [illegible] * | 46" | 50 | 54" | 102" | 102" | | | | | | | |
| Lime Feed Tank Level (Inches)** | 40" | 42" | 42" | 40" | 40" | | | | | | | |
| Gas Meter Reading | | | | | STOP TAKEN | | | | | | | |

TEST FINISHED @ 1335 Hours

* 1 inch = 49 gallons

** 3% $Ca(OH)_2$ by wt., 1 inch = 110 gallons

# APPENDIX H

## CALCULATION OF WASTE DESTRUCTION PERFORMANCE

Waste destruction performance data were calculated for all waste burns. Input into these calculations was taken from several sections of this report indicated in the following examples.

The waste destruction efficiency ($DE_{waste}$) calculation is based upon comparing a waste input rate to a waste emitted rate.

$$DE_{waste} = \frac{\text{waste input} - \text{waste emitted}}{\text{waste input}} \times 100 \qquad (H\text{-}1)$$

Equation (H-1), restated in another form, is

$$DE_{waste} = \frac{I_{waste} - [VFR_{gas}\ E_{waste}]}{I_{waste}} \times 100 \qquad (H\text{-}2)$$

where:

$I_{waste}$ = input rate of organic portion of aqueous waste feed, grams per minute.

$VFR_{gas}$ = volumetric flow rate of combustion gases from the afterburner, dry standard cubic meters per minute.

$E_{waste}$ = concentration of organic waste constituents in combustion gas as determined by GC or GC/ms, grams per dry standard cubic meters.

Similarly the destruction efficiency for total organics ($DE_{total\ organics}$) compares the input rate of combined waste and auxiliary fuel to the emitted rate of all organic material found in the combustion zone samples.

$$DE_{total\ organic} = \frac{I_{fuel} - [VFR_{gas}\ E_{total\ organics}]}{I_{fuel}} \times 100 \qquad (H\text{-}3)$$

where:

$I_{fuel}$ = input rate of organic portion of waste plus auxiliary fuel oil, grams per minute.

$C_{total\ organics}$ = sum of the concentrations of all organics found in the combustion zone samples, grams per dry standard cubic meter.

The calculations for the hammermilled PCB capacitors test are presented below as examples. Initially $I_{waste}$ is calculated, then $VFR_{gas}$, and finally $C_{waste}$ (which in this case is the analytical detection limit) is added to Equation H-2 to calculate $DE_{waste}$:

$$I_{waste} = \frac{210\ \text{kg fluff}}{\text{hr}} \times \frac{0.29\ \text{kg PCB}}{\text{kg fluff}} \times \frac{1000\ \text{g}}{\text{kg}} \times \frac{\text{hr}}{60\ \text{min}} = 1{,}015\ \frac{\text{g PCB}}{\text{min}}$$

$$\begin{aligned}VFR_{gas} = {} & \frac{9.19\ \text{actual meters}}{\text{sec}} \times (1 - 0.306\ \text{mole fraction water}) \times \\ & 3.083\text{m}^2\ \text{cross-sectional stack area} \times \\ & \frac{293^{\circ}\text{K at standard conditions}}{327^{\circ}\text{K at actual conditions}} \times \frac{1.024\text{m}^2\ \text{hot duct area}}{3.083\text{m}^2\ \text{stack area}} \times \\ & \frac{767\text{mm Hg at actual conditions}}{760\text{mm Hg at standard conditions}} \times \frac{60\ \text{sec}}{\text{min}} \\ = {} & 630\ \text{dry standard cubic meters per minute}\end{aligned}$$

$$DE_{waste} = \frac{\left(1{,}015\ \frac{\text{g PCB}}{\text{min}}\right) - (630\ \text{dscm/min})\left(0.005\ \frac{\text{mg}}{\text{m}^3} \times \frac{\text{g}}{1000\ \text{mg}}\right)}{1{,}015\ \frac{\text{g PCB}}{\text{min}}} \times 100$$

The destruction efficiency for total organics, $DE_{total\ organic}$, for the PCB fluff test is calculated in a similar manner using Equation H-3 and inputing the combined waste and auxiliary fuel feed and the concentration of total organics found. The one assumption used in this particular calculation was that the fiber drums were ~50 percent combustible material by weight.

$$I_{\text{total organics}} = \left(\frac{40.18\ \text{liters No. 2 oil}}{\text{min}} \times \frac{840\ \text{g}}{\text{liter}}\right)$$

$$+ \left(\frac{210\ \text{kg fluff}}{\text{hr}} \times \frac{0.29\ \text{kg PCB}}{\text{kg fluff}} \times \frac{1000\ \text{g}}{\text{kg}} \times \frac{\text{hr}}{60\ \text{min}}\right)$$

$$+ \left(\frac{120\ \text{kg drums}}{\text{hr}} \times \frac{0.5\ \text{kg combustibles}}{\text{kg drums}} \times \frac{1000\ \text{g}}{\text{kg}} \times \frac{\text{hr}}{60\ \text{min}}\right)$$

$$= 35{,}800\ \frac{\text{g total organics}}{\text{min}}$$

$$DE_{\text{total organics}} = \frac{\left(35{,}800\ \frac{\text{g total organics}}{\text{min}}\right) - \left[\left(630\ \frac{\text{dscm}}{\text{min}}\right)\left(15.6\ \frac{\text{mg}}{\text{m}^3} \times \frac{\text{g}}{1000\ \text{mg}}\right)\right]}{35{,}800\ \frac{\text{g total organics}}{\text{min}}}$$

$$\times 100$$

$$= 99.97\%$$

uu1467a
HW-122c.5